

# INTERNATIONAL Chemical Engineering and Process Industries

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## Topics of the Month

### The Ministry of Materials

A PROCUREMENT Ministry, the Ministry of Materials, has been set up by the Government under the Lord Privy Seal, Mr. Richard Stokes. The creation of this new department underlines the Government's anxiety over raw materials and prices, the two most urgent economic problems of the day. Although the chemical industry generally will remain under the aegis of the Board of Trade, responsibility for its basic raw materials and some of its basic products will be the concern of the new Ministry. These materials include salt, bromide, calcium carbide, glycerin, molasses, phosphate rock, potash, mercury, arsenic, iodine, bismuth, sulphur and sulphur-bearing materials, sulphuric acid and fertilisers. Speaking in the House of Commons last month, Mr. Stokes said that the last three groups of materials were the most important items under his control; they had not been separated because they were so intermixed that it would be impossible to divide them administratively. In passing, Mr. Stokes said that 18 new plants or conversions for the production of sulphuric acid from pyrites and anhydrite were now planned or under construction and that the first plant should now be operating. By 1955-56 it should have been possible to reduce raw sulphur imports by half compared with present imports.

Iron and steel production would not come under the new Ministry, but it would be concerned with materials for ferrous alloys. Semi-fabricated metals would remain under the Ministry of Supply, non-ferrous and light metals only coming within the scope of the new Ministry, which would take over in its entirety the Non-Ferrous Metals Directorate at Rugby. An important part of the work of the new Ministry would be the conservation of materials, the development of alternative manufacturing methods and the exploitation of new resources.

It is natural to be doubtful of the practical value of yet another addition to the bureaucratic army with which industry must now contend in its everyday problems. It is to be hoped, however, that this concentration of all raw materials problems in one place may eventually result in increased supplies and lower prices.

### The trend of chemical invention

THERE was a slight decline in the number of patent applications last year compared with the two preceding years. There were 31,686 in 1950 as against 33,626 and 33,347 in 1948 and 1949 respectively. These figures are given in the report for 1950 of the Comptroller-General of Patents, Designs and Trade Marks. Discussing the trend

of invention, the Comptroller observes that in the chemical industry there was considerable activity in the production of artificial resins, especially the fibre-forming condensation resins such as nylon; solventless varnishes for impregnating and coating purposes; polythene; polymers of alkylene oxide; resins made from cashew-nut oil; and silicones for use as lubricants and as oils for electrical purposes. There was much interest in the production of synthetic petrols and anti-knock fuels and considerable attention was directed to vitamins, especially the B group, and to antibiotics. The inventive trend in related fields was to such products as germanium and other contact rectifiers for use as substitutes for valve amplifiers, to magnetic cores consisting of ferrites and other oxides such as those of chromium and manganese, and to the use of barium titanates as substitutes for piezo crystals. Luminescent inks for posters, heat pumps for heating buildings and oils seals to replace stuffing boxes were other fields in which the inventor was active.

### Uranium refining

**A**LTHOUGH there has been no confirmation of reports that British, American and Canadian scientists have developed a formula which is as revolutionary in the production of uranium as the cyanide process was in the mining of gold, some interesting developments in uranium refining have recently been the subject of patents. A Swedish method for the recovery of vanadium and uranium from aluminous schists, for example, entails the impregnating of the schist, mixed with solid fuel if necessary, with alkaline earth oxides or carbon, and subsequent granulation and sintering. Treatment with hydrochloric acid is carried out either at the time of sintering or subsequently, and the reaction gas recycled. This yields the volatile chlorides of vanadium and uranium which are removed from the reaction and combustion gases. The metals are afterwards obtained from the chlorides by conventional methods.

In a patented method for the treatment of pitchblende ores, the ore is ground to below 40 mesh, and is then digested with nitric acid to which sodium or ammonium nitrate is added. This dissolves out uranium, radium and lead, and the insoluble matter is filtered off. Sulphuric acid is then added to the solution to cause precipitation of the radium and lead, while the uranium is left in solution. The radium and lead can be separated by treatment with ammonium acetate, which leaches out the lead sulphate.

A third patent constitutes a variation in the electrolytic production of uranium. In this method, one part of uranium is added to three parts of a mixture of alkali and alkaline earth halides at a temperature of about 800°C. Electrolysis is carried out in a graphite tank using an applied p.d. of 6 V.

### Finland's chemical industry

**A**LTHOUGH small by European standards, Finland's chemical industry is firmly based upon indigenous resources of timber and minerals and has flourished for more than half a century. In an average year (*e.g.* 1949) some 1,400,000 tons of cellulose and 380,000,000 boxes of matches are produced, together with 176,000 tons of sulphur concentrates, 21,000 tons of copper and 5,000 tons of zinc concentrates. Fortunately for Finland most of her minerals contain a high proportion of sulphur, so that the current shortage has little effect on her industries. Sulphuric acid has been made in the country for 27 years. There are four

plants operated by three companies. Three use vanadium catalyst, while the fourth uses the chamber process. Total capacity is some 125,000 tons p.a. of 93% sulphuric acid, but output in 1949 was not more than 89,000 tons, although this was 23,000 tons more than in 1948. (Normal U.K. production is 1,800,000 tons p.a.). The paper and pulp industries absorb a great part of the sulphuric acid, but not so much as the fertiliser industry which uses two-thirds of total output to make superphosphates at the rate of some 500,000 tons p.a.

As for minerals, a State-owned organisation works the copper deposits which are the largest, as well as the deposits of arsenic pyrites and tungsten. There are also reserves of zinc, nickel, gold, silver, platinum and selenium.

In addition to the major undertakings based on the exploitation of minerals and timber, there are a number of other chemical companies. Some organic chemicals are manufactured, including cellulose derivatives and solvents. There are three companies manufacturing plastics which include phenol formaldehyde resins, urea and casein-type plastics. In 1949 production of paints and varnishes amounted to 7,000 tons, soap 9,000 tons and washing powders 4,000 tons. Pharmaceuticals are manufactured by two companies.

### Measures to maintain rayon output

**I**T is remarkable how resilience, determination and improvisation can help an industry through a crisis. When the full implications of the sulphur shortage dawned upon us eight months ago, one of the industries which we all imagined slowing quickly to half speed was the rayon industry since, of course, sulphuric acid and carbon bisulphide are essential to its operation. In the event, during the first quarter of the year, total production of filament yarn and staple fibre fell by only 10%, compared with the last quarter of 1950. That the fall was not greater was due to immediate measures which the industry took to conserve these chemicals, to the temporary effect of drawing on such stocks as were to hand, and to the maintenance of production of those man-made fibres requiring little sulphur. The producers also co-operated with the Government in reducing exports of rayon both in yarn and staple fibre form. Steps were taken to secure supplies of staple fibres from abroad and stocks of both yarn and staple fibre were run down. Although the full effect of some of these measures will not be felt till later in the year, the result was that total deliveries of yarn and staple fibre to industry in the first quarter of 1951 were only 4½% below what they were in the last quarter of 1950. The producers also showed great enterprise in securing additional supplies of sulphur, carbon bisulphide and even sulphuric acid from abroad. The quantities were only marginal, but they have made it possible to increase production once more. The combined effect of all these measures should be at least to maintain supplies to the yarn- and fibre-using industries.

In congratulating the industry on this achievement at a luncheon of the British Rayon Federation in Bradford last month, the Parliamentary Secretary to the Board of Trade emphasised that we were far from overcoming the sulphur shortage as yet. Everything possible must be done to accelerate the construction of the new plants to produce sulphuric acid from pyrites and anhydrite. Not until these plants are working can the rayon and the many other industries depending upon sulphuric acid feel reasonably confident of their needs being met.

## Sulphur in history

IT is a human tendency to imagine that immediate contemporary problems are unique in their size and complexity and to forget that often they are simply a recurrence of difficulties which have been faced and overcome in the past. A study of a particular problem in history sometimes helps in overcoming it in its contemporary form and, no doubt, it was with this in mind that for his presidential address to the Society of Chemical Industry at the annual meeting in London last month Mr. Stanley Robson chose the subject of sulphur. In his wide-ranging and authoritative survey, Mr. Robson showed that sulphur supplies have periodically caused anxiety for well over a hundred years. The early British chemical manufacturers used Sicilian brimstone almost exclusively and were deeply disturbed when in 1838 the king of Sicily granted a marketing monopoly to a French concern, with the result that prices were raised alarmingly. This caused manufacturers to turn their attention to alternative sources of sulphur and in 1839 some 18 patents for pyrites-burning processes were filed in Britain. The conversion from brimstone to pyrites proceeded steadily from then onwards and by 1860 pyrites completely dominated the sulphuric acid industry in Great Britain and it seemed that there would never be a return to brimstone for acid manufacture.

The technological revolution which confounded this belief and which brought us to our present dependence upon American brimstone was Herman Frasch's process for the superheated steam extraction of sulphur from the domes of the Texas Gulf area. This process which led to the production of brimstone at unheard of low prices was first commercially operated by the Union Sulphur Co. in 1903. In their first year, Union Sulphur extracted 190,000 tons of brimstone by the Frasch process. By 1914 output had risen to 418,000 tons p.a., compared with 372,000 tons p.a. in Sicily. By 1918 some 1,350,000 tons p.a. were being extracted from the Gulf sulphur domes and thenceforth the Sicilian industry steadily declined in the face of vigorous American competition.

Now the wheel has turned full circle and we are back to the situation of more than 100 years ago when we had to convert our acid-making plants from brimstone to pyrites. During the past 50 years the world has gobbled up its easily won supplies of natural sulphur and must now make increasing use of less readily available sources. There are at least 400,000,000 tons of pyritic ores awaiting exploitation, compared with 55,000,000 tons of American brimstone, enough for only ten years at the present rate of use.

Mr. Robson, an authority on sulphur and sulphuric acid, believes that much greater quantities of sulphur should be obtained from metallurgical processes. The object must be to extract it in the form of easily stored and readily handled brimstone. Gas taken from a roasting plant and converted direct to sulphur would be the simplest way of extracting the element from metallurgical processes. Natural gas is another, although lesser, source of sulphur; there is a potential of some 1,000,000 tons p.a. from natural gas. Anhydrite, already used by I.C.I., is another source which is being actively developed in this country. Finally, there is the possibility of producing sulphur as a by-product of oil refining and, in this connection, there is the recent announcement of the Esso Petroleum Co. of a project to produce 12,000 tons of sulphur p.a. from the operations at its new refinery at Fawley which went into partial production last month.

## France tackles the sulphur shortage

THE setting up of facilities for the extraction of sulphur from Spanish pyrites in the Toulouse region, together with an increase in output from the deposits of Languedoc and Provence, should enable France to balance her sulphur needs and resources by 1953, and even allow a margin of sulphuric acid for export. It is hoped that France will become less dependent on imports through the application of the policy of economy and recovery laid down by the Government. Official circles estimate that annual recovery could reach 180,000 tons, 140,000 tons of which would be extracted from the 14,000,000 tons of petroleum processed each year in French refineries. The remainder would come from coking plants (15,000 tons), the viscose industry and the sulphuric acid industry.

Until recently, France had not felt the effects of the world sulphur shortage. Her main sources of supply are her blende and pyrites roasting plants, sulphur extractors in the petroleum refineries, and a few mines—the balance being made up by imports from the U.S.A. and Italy. However, the shortage will now be felt, since requirements, which were estimated at 170,000 tons at the beginning of this year, are actually higher, and imports from the U.S. and Italy will probably not reach the tonnages foreseen. Sulphur needs for 1951 are now estimated at 190,000 tons. Of this amount, 40% will go to agriculture, 50% to the production of sulphuric acid, carbon bisulphide and other sulphur derivatives and the rest to industry (dye manufacture, explosives, rubber, etc.). These requirements are expected to increase to 250,000 tons within two or three years.

## Vapour compression distillation

'WATER in industry' was the theme of the lectures at this year's annual meeting of the Society of Chemical Industry. One paper of chemical engineering interest was that given by J. Leicester of the Admiralty Materials Laboratory on the vapour compression still. Mr. Leicester recalled that the plant embodies no new principle but that the idea was revived during the last war by Dr. Kleinschmidt in the U.S., and a large number of mobile plants were constructed.

This evaporator has often been quoted as 'eating its own tail.' In point of fact, this does happen once boiling of the liquor has been started by the supply of a small amount of external heat. Low-pressure vapour from the boiling liquid is removed, compressed, and re-enters the heating coils of the evaporator to provide the heat source for further evaporation. Practically the whole of the latent heat of the vapour is used in heating and evaporating the liquid product. Extensive heat exchangers are fitted to retain as much heat within the system as possible. Also, if the unit is directly driven by an internal combustion engine, heat exchangers are provided to remove engine jacket and exhaust waste heat.

Scale formation on the heating surfaces has an adverse effect on the efficient performance of a vapour compression still. In the case of stills operating by electrical power, scale is a more serious factor than in those driven by an internal combustion engine. In the former, complete failure of scaled heating elements can occur, whereas, in the latter, only economy of operation suffers. Developed techniques for the prevention of scaling in conventional design evaporators have shown considerable promise for application in compression stills.

Economy of operation of compression stills shows a

remarkable saving in cost when compared with a conventional evaporator. In an electrically driven plant, assuming an overall mechanical and electrical efficiency for the drive of at least 65%, at least 120 lb. of water can be produced for the expenditure of 1 lb. of fuel. Several modern American plants using direct diesel-driven compressors and exhaust waste heat regenerators produce as high a figure as 270 lb. of water per lb. of fuel consumed. These figures contrast with 9 lb. of water per lb. of fuel consumed in the conventional single-effect evaporator.

### Heavy water as a research tool

ANOTHER paper in the S.C.I. series on 'Water in industry' was on the use of heavy water as a research tool. It was given by Prof. H. S. Taylor of Princeton University, U.S.A., who began by pointing out that heavy water became the first isotopic molecule available to industry in tonnage quantities in which a single isotopic species of one of the elementary constituents was present. The electrolysis of water combined with a catalytic process of stripping the deuterium content of the evolved hydrogen through exchange reaction with water vapour and return of the deuterium-enriched water to the electrolytic system made the industrial production of deuterium oxide a possibility. The major portion of the heavy water produced is employed as a moderator in nuclear reactors. It is a more effective moderator than graphite in slowing down neutrons and has a smaller neutron absorption. It has permitted the development of high-powered reactors relatively small in size.

Deuterium is the most generally useful of the stable isotopes, though there are limitations on its use. Industry is now supplying some dozens of inorganic and organic deuterated compounds, under the control of the Atomic Energy Commission in the U.S.A. Concentrations in excess of 95% are possible in the deuterium-labelled positions.

Such compounds have become the tools in a wide variety of scientific research and industrial application. They were earliest employed in exploration of catalysis at surfaces and yielded immediate advances in this field. Especially in the petroleum industry deuterium has contributed significantly to our knowledge of hydrocarbon reactions, more recently with the use of  $H^2$  and  $C^{13}$  for 'double labelling.' Applications in the biological field led also to increases in our knowledge.

More recently the importance of deuterated compounds as an industrial analytical tool, making use of the mass isotope dilution method, combined with infra-red absorption spectra as the measuring index, has been emphasised. As examples from the recent literature, the determinations of the gamma-isomer of benzene hexachloride, of nicotinic acid and of penicillin assays in fermentation were mentioned.

### Chemical oceanography

THE need, in a world of dwindling raw material reserves, to exploit the chemical resources of the sea has been emphasised before in these notes. We pointed out that it would be appropriate for this country, comparatively poor in natural resources, to initiate full-scale development of the resources of the sea. That the need for such development is urgent is underlined by the news that in America, rich in natural resources, active study of the chemical potential of the oceans has been started. Chemical oceanography is the name that has been coined for this new branch

of applied science and a Chair bearing this name has been established at the Texas A. and M. College. The first Professor of Chemical Oceanography in the U.S. is Dr. Donald W. Hood.

Although man became interested in marine chemistry as long ago as 2000 B.C., when Romans, Greeks and Egyptians extracted salt from sea water, Dr. Hood points out that chemical oceanography is far wider in scope than marine chemistry as known today. Marine chemistry is concerned with the composition and distribution of the chemical constituents and the chemical properties of the sea. Among the problems which chemical oceanographers (there are only about 30 in the whole world so far) should study, according to Dr. Hood, are:

The rate and mechanism of decontamination of polluted waters at the mouths of rivers; the osmotic relationship of fresh-water fish which spend part of their life cycle in the sea; the nutritive value of the tiny sea organisms called plankton, and an economical culture method for these organisms; the chemistry and extraction of trace elements from sea water; the study of chemical sedimentation; the mechanism of the microbiological reactions involved in corrosion, formation of petroleum, and so forth; and extraction of important pharmaceuticals from marine organisms.

### Parkes, Pattinson and desilverisation

JUST a century ago Alexander Parkes, that versatile inventor of Birmingham, developed and patented his well-known process for the desilverisation of lead. He had taken out a preliminary patent the year before. We see today how 1851 was the point in chemical history where Parkes outbid what Hugh Lee Pattinson had accomplished with his desilverising process.

The two men came to different processes by equally different routes. Parkes had little schooling; he was early an apprentice in electroplating at Elkingtons. But there it was that he proved himself the practical chemist and inventor, both regarding metals and in other chemical fields. To coat fragile flowers or spiders' webs with a fine deposit of silver before electroplating them; such was one example of the work of Parkes, styling himself 'artist' at first, gradually becoming chemist. To reduce silver nitrate to the silver deposit brought his use of phosphorus dissolved in carbon disulphide. So it followed that Parkes turned to improve the manufacture of carbon disulphide, then selling at two guineas a lb. Soon he enabled it to be sold at 2s. a lb.; then went on to produce sulphur chloride when both solvents were needed for his waterproofing process which he sold to Macintosh & Co. in 1841.

While Parkes began as an electroplater's assistant and arrived at his desilverising process, Pattinson seemed more set for such an invention. Though a soap-boiler's apprentice at first, he became Cumberland metallurgical chemist and assay-master to the Alston mines. His desilverising came earlier than that of Parkes, Pattinson being manager of a northern lead works at the time. He manufactured white lead and magnesia alba and established a chemical works at Washington, near Gateshead. Yet although with *pattinsonieren* in German and *pattinsonage* in French his name seemed destined for perpetuation, Pattinson's process had to give way to that of Parkes which began at Llanelli and became the standard operation in Germany and the United States.

# Vacuum Refrigeration

By D. Wittenberg

Vacuum refrigeration, or steam jet refrigeration, has several advantages over chemical methods of producing cold. It is simple in construction and operation and is comparatively cheap to install and use. It can readily be automatically controlled, requires little space and can be installed in the open. It has several applications in the process industries, including the production of crystals, and large units are in operation in different parts of the world. The theory of vacuum refrigeration, the construction of commercial plants and their applications are described in this article.

ALL liquids have vapour pressures. When the vapour pressure reaches the surrounding gas pressure the liquid boils. Thus, if the pressure of the air in contact with a liquid is reduced, the liquid will boil at a lower temperature than the boiling point at normal atmospheric pressure. This is the first factor to be considered in vacuum refrigeration. The next factor is that if the vapour of the boiling liquid is drawn away from the bulk of the liquid and condensed elsewhere, then the temperature of the liquid will fall owing to the removal of a quantity of heat from the liquid proportional to the weight of the vapour and the latent heat of vaporisation of the liquid.

Thus by maintaining in a closed vessel a pressure equal to the vapour pressure of the liquid at the desired temperature, and by continuously pumping away the vapour, a refrigeration system may be achieved.

## Development of vacuum refrigeration

The principle of this process was well known in the early part of this century, but it was not applied successfully. Therefore it was of more theoretical than practical interest.

That this refrigeration method was not successfully used until 15 to 20 years ago was due mostly to lack of wide knowledge of the results obtained by manufacturers during long and expensive experiments, and to the fact that the refrigeration industry developed around the production of sub-freezing temperatures by means of chemicals. It is only recently, with the demand for refrigeration at temperatures above 32°F. for air conditioning and industrial process cooling, that the water vapour refrigeration cycle has become commercially attractive. Another, perhaps the most serious, hindrance to the development of vacuum refrigeration was that it depended upon conventional reciprocating vacuum pumps to produce the vacuum and draw off the generated vapour until recent improvements in the steam jet vacuum producer and steam booster provided economical equipment for the practical application of the method. Steam jet equipment manufacturers were quick to appreciate the possibilities of this new market and during 20 years of continuous research and development have succeeded in perfecting today's efficient vacuum refrigeration units.

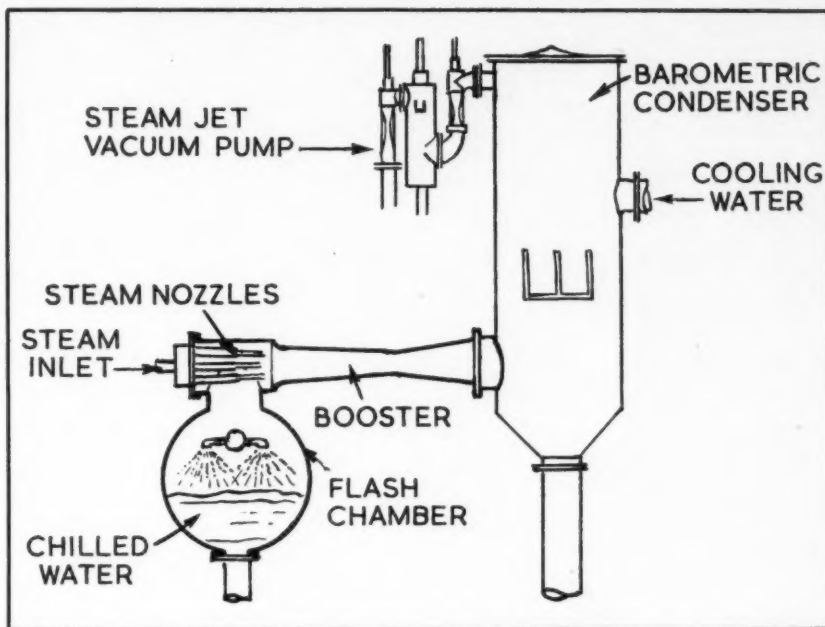


Fig. 1. Steam jet booster and vacuum refrigeration unit.

## The steam jet vacuum producer

This device can be understood if we consider the problem of producing 30,000 lb. hr. chilled water at 45°F. from water made available at 70°F. This will require a total heat absorption of 750,000 B.Th.U. and a vacuum corresponding to an absolute pressure of 0.3 in Hg. At this pressure the latent heat of evaporation of water is 1,066 B.Th.U./lb. Dividing 750,000 by 1,066 gives 700 lb. hr. of water which must be evaporated and removed at the rate of 1,745,000 cu. ft./hr. (specific volume of the water vapour at 45°F. is 2,033 cu. ft.) about 29,000 cu. ft./min. It is obvious that a great number of reciprocating vacuum pumps would be required for this duty. By contrast, the steam jet vacuum producer, operating at high velocities on an entrainment rather than a displacement principle, can handle this capacity in a single medium size unit which weighs, with condenser, about 4,000 lb., and through which the steam and entrained vapour attain high velocities.

## Typical units

Vacuum refrigeration units with baro-

metric condensers are used where condensate recovery is not necessary. Such units are generally installed outdoors and require only a simple foundation. Operating steam issuing at high velocity from the nozzles of the steam jet booster entrains and removes the large volumes of water vapour constantly forming in the evaporator, thereby maintaining the high vacuum necessary to cause the evaporation at the desired chilled water temperature. The mixture of operating steam and water vapour is compressed and discharged from the booster into the condenser where it is liquefied or condensed by contact with cooling water. Where condensate recovery is essential, a surface condenser is used. In this type the condensate is removed by a pump and fed back to the boilers or discharged elsewhere as desired. The water chilled in the evaporator, as described, is removed by a centrifugal pump and delivered to the point of use. Here it absorbs heat and is returned to the refrigerating system for cooling. A steam jet ejector or vacuum pump maintains the condenser vacuum while the unit is in operation by removing air and any non-

condensables which leak into the system. In starting the unit, the ejector produces the initial vacuum by removing air from all parts of the equipment before the boosters are put into operation.

### Steam jet booster

As mentioned, it was mainly due to the perfection attained in the design and construction of the steam jet booster that vacuum refrigeration was successfully applied. This equipment consists essentially of several steam nozzles discharging jets of steam at velocities depending on the steam pressure and the pressure at the outlet of the nozzles, and attains velocities up to 6,000 ft./sec. across the suction chamber (Fig. 1) and through a venturi-shaped compression tube. Water vapour enters the booster suction and is entrained by the jets of steam and discharged through the throat and diffuser where the velocity or kinetic energy is converted into pressure. The water vapour is thus compressed and discharged into the condenser at a higher absolute pressure than that maintained in the evaporator. Experimental research forms the background of booster design. The proportions, size and arrangement of the essential parts have a vital influence on the capacity and compression range.

### Control

The capacity of the steam jet booster, and with it the capacity of the vacuum refrigeration unit, cannot be effectively controlled by varying the flow of steam. The steam jet boosters must generally be turned on completely. Throttling of the steam supply does not directly reduce the booster's capacity, but instead materially affects its ability to compress sufficiently to discharge into the condenser. Control of the capacity of the refrigeration unit is generally taken care of by turning on or turning off more boosters, using manual or automatic control, as required.

### Capacity

The capacity of a given vacuum cooler expressed in tons of refrigeration (1 ton of refrigeration is equal to 12,000 B.Th.U./hr.) varies widely with the temperature at which the refrigeration effect must be pro-

duced. Steam jet boosters can be constructed to operate successfully with steam pressures from 2 lb. gauge and upward. The lower the steam pressure, the greater the amount of steam and condenser water required. Steam boosters must be designed and built for the lowest steam pressure at which they operate.

The maximum absolute pressure without water in the chilling tank attained up to date, is slightly lower than 1 mm. Hg., to which corresponds a water temperature of about 3°F. When operating at a vacuum sufficiently high to form ice, the steam consumption of the jets is such that operation costs are usually higher than for mechanical refrigeration. However, low cost steam and relatively cold condenser water in some cases make the steam jet unit an economical proposition even at temperatures a few degrees below freezing.

### Advantages

Although steam jet refrigeration requires more cooling water than chemical refrigeration it has the following advantages when compared with other systems:

- Comparatively low cost of installation and operation particularly when waste low-pressure steam is used and the condenser cooling water is re-used for other purposes.
- It is of simple design and construction and contains no moving parts except the pumps. Thus low maintenance cost and practically no supervision is required.
- It can readily be automatically controlled and is easily adjustable for the load, responding quickly to load variation, and has high overload capacity with slight increase in cold water temperature.
- It requires little space and can be installed outdoors. It is self-supporting and does not require any additional construction (see Table I).

### Applications

Steam jet vacuum refrigeration is suitable for producing drinking water and chilled water for air conditioning, for increasing absorption in chemical plants and sulphite pulp mills, for cooling warm condenser water and for obtaining adequate

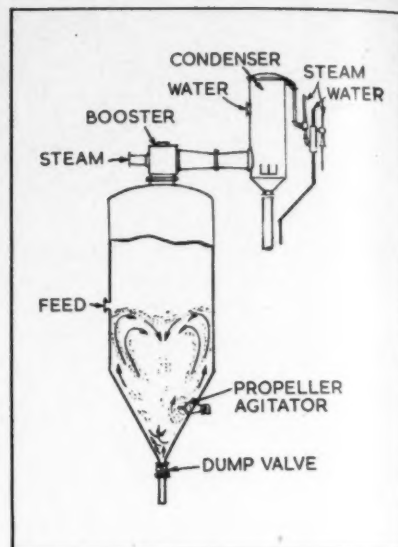


Fig. 2. Vacuum refrigeration crystalliser for batch operation.

fractionation operation in the process field. There are several units of more than 5,000 tons in operation in the U.S. Many vacuum refrigeration units have been installed for cooling water in breweries and distilleries, and have been used to cool, by direct evaporation in a flash chamber, solutions containing great quantities of water in the chemical and food processing industries. Vacuum refrigeration is used with success in cooling sugar, starch, caustic solutions and yeast. Its advantage in such cases is that there is no need for transfer surfaces which usually foul the products and hinder heat transfer. Vacuum refrigeration is used on ships for air conditioning and for cooling drinking water. It requires little space and is free of vibration and noise.

### Batch cooling

Batch cooling is used particularly where the cooling range is 100°F. and more. Generally, the batch method is more efficient than the continuous method, as it permits most of the vapours to be evacuated from the evaporating chamber at a higher mean temperature. Evaporation starts with the temperature of the liquid entering the vacuum chamber and it is at this point that the steam jet attains its maximum efficiency. The efficiency drops as the temperature in the vacuum chamber approaches its final value. In the continuous method the steam jet has to maintain continuously the maximum vacuum corresponding to the lowest desirable temperature and, therefore, operates at its lowest efficiency.

Batch operation is equivalent to the integrated sum of an infinite number of continuous stages. In batch operation, the temperature and absolute pressure is gradually decreased. When the booster is first turned on, the pressure range

TABLE I

Tons of Refrig. at 50°F.	Approximate Dimensions					
	Barometric Condenser unit			Surface Condenser unit		
	width	length	height	width	length	height
	ft. ins.	ft. ins.	ft. ins.	ft. ins.	ft. ins.	ft. ins.
20 .. .. .	6 0	10 0	45 0	6 0	10 0	9 0
40 .. .. .	7 0	10 0	45 0	9 6	12 6	10 0
50 .. .. .	8 0	11 0	46 6	10 0	12 6	10 0
75 .. .. .	9 0	11 0	46 6	12 0	12 6	11 0
100 .. .. .	10 0	12 0	47 6	12 6	15 0	11 0
125 .. .. .	11 0	12 6	47 6	13 0	17 0	11 6
150 .. .. .	11 0	13 0	51 0	14 0	17 0	11 6
200 .. .. .	12 0	14 0	53 0	15 0	18 0	13 0
300 .. .. .	12 0	16 0	54 0	18 9	20 0	15 0

1 ton refrigeration = 12,000 B.Th.U. per hr.

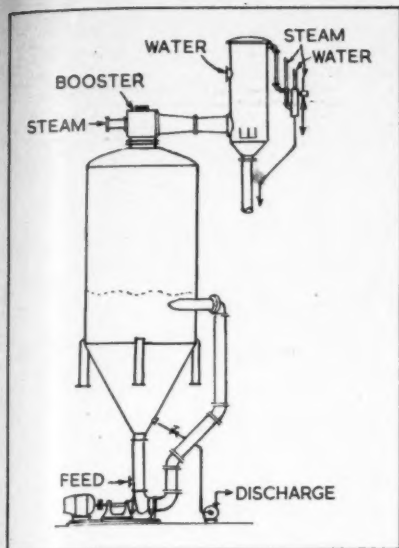


Fig. 3. Vacuum refrigeration crystalliser for continuous operation.

through which the vapours must be compressed is very small and the booster's capacity to handle vapour is relatively high. The compression range increases as the process cycle continues so that at the end temperature and lowest absolute pressure the compression range of the booster is at its maximum, and its capacity to handle vapour is the lowest. The average capacity of the booster operating over the complete cooling range in a batch vacuum refrigeration unit is thus greater and more efficient regarding steam and cooling water consumption, than in a single stage continuous vacuum refrigerator. Batch operation is used when the quantity of the liquid to be cooled is moderate and when the temperature is relatively low.

### Continuous operation

When large quantities of solutions or liquids are to be cooled (over 200,000 gal./day) the lower steam and cooling water consumption for batch operation is usually offset by the higher installation cost. Thus, for large quantities multi-stage continuous vacuum refrigeration is generally used. As the number of stages of continuous refrigeration units in series increases the overall performance approaches the economy of batch operation.

### Vacuum crystallisation

Vacuum refrigeration is an advantageous method of removing heat from a solution for the formation of crystals. Besides the advantages mentioned above, it involves lower first cost than mechanical crystallisation and, it is claimed, produces a more uniform crystal. Vacuum crystallisers are usually provided with vertical and horizontal agitators to agitate the solution and to keep the crystals in suspension. Vacuum crystallisation by vacuum refrigeration can be successfully applied to batch or con-

tinuous operation and to single, multi-stage or compound units (see Figs. 2 and 3) depending on the volume of solution to be handled and its chemical and physical properties.

In the batch crystalliser a charge of hot concentrated solution is introduced into the vessel in such a way that it reaches the surface of the liquid in the crystalliser. The solution, having a definite boiling temperature at the vacua existing in the vessel, will spontaneously and adiabatically cool and crystallise (if the solubility of the solute decreases with the decrease of temperature) because of the evaporation of a part of the solvent. In cases where the cooling water is too warm to condense the vapours, a steam jet booster, as described above, is used to compress them to a point where they can be condensed by the cooling water available.

In the continuous single stage vacuum crystalliser the solution is fed continuously into the vessel in which the highest vacuum exists all the time and the cooled solution or slurry is continuously pumped out, maintaining a constant level of liquor in the crystalliser. In the multi-stage crystalliser the solution is fed from one crystalliser to the next, generally without separating the crystals from the solution between stages.

In addition to the advantages mentioned for vacuum refrigeration crystallisation by this method has these additional advantages:

- (a) Crystallisation occurs by simultaneous evaporation and cooling.
- (b) Running costs are comparatively low.
- (c) Rubber- and lead-lined equipment may be used for handling corrosive solutions.

Disadvantages are:

- (a) More cooling water is required than in mechanical type crystallisation.
- (b) Considerable headroom is necessary.
- (c) There is a practical limit on cooling which depends on the boiling point elevation of the final mother liquor.

### Centrifugal and axial flow compressors

Today the design and construction of centrifugal and axial flow compressors has attained a high degree of perfection, and they are built in capacities up to 100,000 cu. ft. min. with a maximum adiabatic efficiency of 87% and at speeds attaining 20,000 r.p.m. The use of such a machine instead of a steam jet booster may be considered, particularly where steam is not available at convenient prices and electric power is cheap.

### Cooling liquids for the condenser other than water

As mentioned, one of the disadvantages of vacuum refrigeration and crystallisation is that it requires relatively large quantities of cooling water for condensing the vapours. This disadvantage increases with the temperature of the cooling water

and as a result reduces the efficiency and capacity of the steam booster. It often happens in chemical processing that concentrated brine solutions and mother liquors are available in great quantities and are usually wasted. Generally these liquids, due to their great specific gravity, have lower vapour pressures than water and can be used successfully as cooling media instead of water. As a matter of fact, the purpose of the steam booster in vacuum refrigeration and crystallisation is to compress the low temperature vapours to a pressure in the condenser high enough for the available cooling water effectively to condense them. It is obvious that if a brine or liquor is used as cooling media in the condenser it will have greater effect if its vapour pressure is lower than that of water at the same temperature. The compression range of the booster will be smaller and thus its steam consumption less. At the Dead Sea where cooling water is not available, vacuum refrigeration can be applied with great success thanks to the fact that brines and mother liquors, having very low vapour pressures, are available in considerable quantities.

## New standards

### Colour identification of pipe lines.

During the past 20 years three British Standards have been published dealing with the identification of piping, and certain anomalies have arisen. In order to remove these difficulties, and to co-ordinate requirements, this code has been prepared and replaces B.S.457—'Identification of chemical pipe lines,' B.S.617—'Identification of pipes, conduits, ducts and cables in buildings,' and B.S.3011—'Identification colours for engine-room piping,' but it does not supersede the British Standards dealing with gas cylinders and medical gas cylinders nor does it affect the provisions of B.S.158, which deals with the colour identification of switchgear bus-bars and connections and wire. Coloured illustrations of the application of the code are included. (Price 2s. 6d.)

**Thermodynamic properties of refrigerants.** B.S.1725:1951 gives tables of thermodynamic properties of the following refrigerants: ammonia, sulphur dioxide, carbon dioxide, methyl chloride and dichlorodifluoromethane. Tables of the properties of saturated vapour and superheated vapour for each of the above refrigerants are included in this standard. The tables of the properties of ammonia, methyl chloride, sulphur dioxide and dichlorodifluoromethane have been reproduced from data published in various journals in the United States of America. Tables of properties of carbon dioxide have been obtained from sources in this country. These tables are reproduced by permission of the various authorities and acknowledgment to them is given in the introduction to the standard. (Price 10s. 6d.)

## CENTRIFUGING

### New machines, auxiliary systems and industrial applications

By E. Broadwell

SINCE the previous review of centrifugation in this journal,<sup>1</sup> two further surveys have been published,<sup>2,3</sup> the former briefly covers many general points, whilst the latter relates specifically to the machines manufactured by the Sharples Corporation.

Progress in fundamental studies is reflected in only one article which deals with the critical wetting rate of various types of cake formed in a perforated basket-type centrifuge.<sup>4</sup> A technique was developed using a photoelectric detector whereby the appearance of diffuse reflection from a dry cake was used to judge the drainage rate at the critical conditions. From the results the history of the cake formation and compressive force applied were found to be of paramount importance when elucidating the hydroextraction properties of the materials under test; it was also established that the cake formation is closely connected with the flexibility of the backing cloth and supporting gauze as these appreciably affect the permeability of the cake. Due to the difference in cake formation, lack of reproducibility is to be expected and it is suggested that further study be given to this problem.

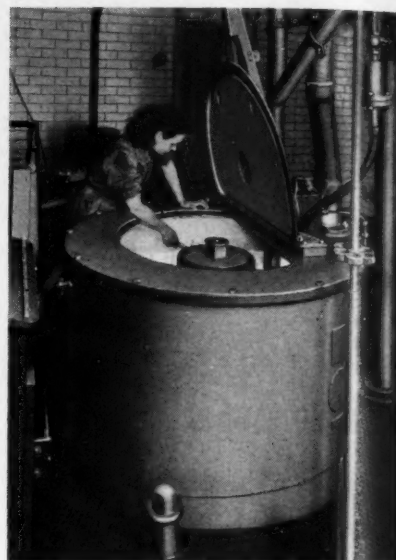
#### Laboratory machines and uses

Several new or improved types of discontinuous centrifuges suitable for laboratory work have been announced. One such machine is designed so that the tubes adjust themselves in the direction of the resultant centrifugal force; to reduce the strain on the glass tubes during rapid acceleration the tubes swing in the opposite direction to that of the rotation.<sup>5</sup> Another machine capable of speeds up to 5,000 r.p.m. and having a six-tube fixed-angle head has been described.<sup>6</sup> The salient feature of a recent patent provides means for accurately regulating the uniform interior heating of a machine suitable for the determination of butterfat in milk.<sup>7</sup> An improved type of apparatus suitable for obtaining ultrafiltration centrifugally, using a Cellophane bag as membrane, is described in detail.<sup>8</sup>

A useful account of the rapid determination of fat in the plant control of cocoa products shows the value and accuracy of centrifugal means of analysis.<sup>9</sup> It is stated that results by this method compare very favourably with the official method of the A.O.A.C. The development of a rapid centrifugal method for the estimation of acetone foots in raw linseed oil is also in this category. This employs a specially designed tube which is centrifuged under specified conditions. Comparison is made between the results obtained by this method and that recommended by the A.S.T.M., from which it is concluded that the former gives reproducibility equal to, or better than, the latter.<sup>10</sup> Centrifuging has also been proved useful as a laboratory method for separating hydrocarbons in a pure form in the fractionation of petroleum.<sup>11</sup>

**New industrial machines**

An interesting development is the Luwesta extractor which was displayed at the recentACHEMA Exhibition in Frankfurt.<sup>12</sup> This machine consists of three sets of discs separated by barriers in a single bowl shell, with mixing and separating devices for each set. It is claimed that with such an arrangement it is possible to effect a three-stage countercurrent extraction of a liquid by a solvent, although no operating data has been published as yet. The display by Podbielniak, Inc., at the Chicago exhibition last September also brought to light new developments in continuous countercurrent centrifugal machines for liquid-liquid extraction. It is reported<sup>13</sup> that not only are these



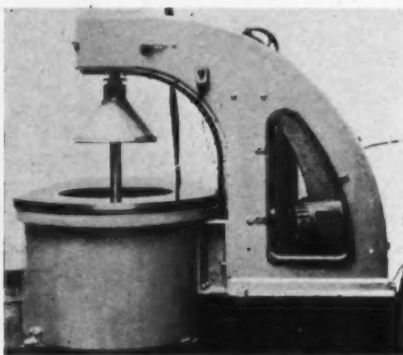
[Photo: Thomas Broadbent & Sons Ltd.]

This direct-electrically under-driven centrifuge with bottom discharge is used for separating a solution of fine crystalline salt and liquor. The solution is continuously fed into the basket while running at half speed and when a thick cake of salt has built up in the basket interior the centrifuge is run at a full speed, the speed is then reduced by half, the cake washed off and the centrifuge re-run at full speed. The solid mass of salt is then broken down and discharged through the bottom of the basket. The perforated basket, basket linings, feed and wash pipes, fume outlet and discharge are all made of stainless steel.

machines capable of handling liquids at capacities ranging from 500 c.c.s. per minute to 20,000 gal./hr., but models are now available for handling liquids containing suspended solids, such as the broths obtained in the production of antibiotics, in which the liquid is extracted and the solids washed in two stages.

A claim for a centrifugal separator suitable for the classification of granular solids of different densities in a liquid medium, has been put forward.<sup>14</sup> This is suitable for the separation of such materials as minerals in which particles of 100 mesh or finer may be obtained; the machine consists of a tapered cylindrical shell and two worms which feed the different fractions to their respective outlets after separation. A new underdriven basket machine capable of taking an 84 lb. load and running at 700 r.p.m. has been announced<sup>15</sup>; it reaches top speed in 4 sec., is braked electrically in 3 sec., and is provided with a cover interlock. One of the interesting features of a recent patent covering a centrifugal cream separator is that the bowl may be effectively washed inside and out without dismantling the separator.<sup>16</sup>

A number of other new industrial



[Photo: Watson, Laidlaw & Co. Ltd.]

A 48 x 24 in. electrically driven suspended centrifuge having a perforated Monel metal basket to run at a speed of 750 r.p.m. Other parts include an aluminium central bottom discharge valve (in halves) and Monel metal basket linings, mild steel monitor case with fume gutter at top (lined inside with chemical sheet lead), wood cover in halves, solid steel spindle sheathed with pure nickel tube and cast iron framing and soleplate.



(Photo: Alfa-Laval Co. Ltd.)

Typical Beer Wort clarifying and cooling installation using a De Laval heat exchanger and clarifier.

machines have been described, but their use relates specifically to the sugar industry, and are dealt with in a later section.

#### Auxiliaries and control systems

With the increasing use of centrifuges in industry there is a trend to utilise more automatic controls and auxiliary equipment to achieve greater efficiency and to eliminate the human element. Although some of the devices have been developed for very specialised purposes, by suitable adaptation and modification other applications are possible.

A solution to the following problem has been published<sup>17</sup>: the torque measured by the controller of a conveyor, used for removing salt from the basket of a machine separating a glycerine/salt slurry, depends upon the salt concentration. If the salt concentration was low the feeding arrangements were such that overloading of the motor would result; this was overcome by installing pneumatic relays in the feed control system so that they were responsive to current load on the centrifuge motor, torque on the conveyor and level of the filtrate in the receiving tank.

In the case of a Bird machine separating fine coal slurries, a control has been devised which prevents overloading of the machine, first by stopping the feed and then the machine, should the overloading still continue to increase.<sup>18</sup>

Other inventions which have been described for use with basket-type centrifuges are: a separate motor with interlocking electric clutch used for discharging solids<sup>19</sup>; a means for controlling the speed of a centrifuge motor at low speeds when discharging<sup>20</sup>; a safety device for locking the mechanism of a discharging shoe and assembly<sup>21</sup>; a discharging mechanism comprising a guide housing, discharger shaft

and shoe<sup>22</sup>; automatic washing of solids with special nozzles<sup>19</sup>; a solenoid controlled three-port valve working in conjunction with spray nozzles<sup>23</sup>; automatic cycle control employing timer elements connected to a two-speed wound motor and braking controls<sup>24</sup> and air-operated gates for charging centrifuge baskets.<sup>19</sup>

#### Sugar

New machines announced for use in this industry show several improvements over existing types; among these the Hepworth hydraulic centrifugal has been described in some detail.<sup>25</sup> It is capable of speeds up to 1,800 r.p.m., has rapid acceleration and is completely safe in that it cannot overspeed. The chief feature is that large and small impulse wheels are utilised in a water motor; the operation of this motor is clearly described with graphs. Details of the performance in practice are given and from the power consumption standpoint, it is stated that the drive employed in these machines compares favourably with conventional types of drive.

A new approach to centrifugal driers makes use of one or more filtering membranes arranged in stages at or about right angles to the axis of rotation in a bowl shaped like a truncated cone.<sup>26</sup> It is claimed that the products being dried in such a machine will not have a tendency to clog the membrane, uninterrupted operation is ensured, and by using a scraping mechanism successive transfer of the dried material from one filter to the next until it is finally discharged over the weir of the last filter is possible. Another patent makes use of a continuously moving belt-like filter surface carrying the material through washing and filtering zones, thus avoiding the need of mechanical scrapers with resultant loss of solids.<sup>27</sup>

An abstract from a paper by Broadbent dealing with machines used in British sugar refineries reviews present practice in this industry.<sup>28</sup> This includes types of drive, means of charging and discharging the machines, automatic controls, and suggestions for collaboration regarding further developments. Another article dealing with Jamaican practice mentions advances in the purging of massecuites and modern trends of design, which includes flexible coupling or hydraulic fluid drive, water-cooled regenerative braking, and push-button automatic controls for charging and discharging.<sup>19</sup> Information on the successful performance of Roberts centrifugals in various sugar producing areas is disclosed<sup>29</sup>; low speeds via belt and gears are provided by one motor driving a group of machines; as only one machine is started at a time, the peaks on the motors are reduced in proportion to the number of machines. In a review of some aspects of the remelt process and the centrifuging of B massecuites, opinions regarding the unsuitability of the former are expressed.<sup>30</sup>

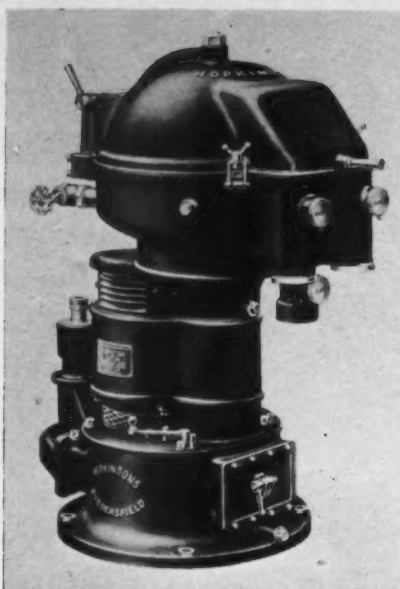
Other references to the centrifugal treatment of sugars include: information obtained by clarifying syrup sulphited to pH 6 so as to remove a greater proportion of non-sugars; clarification of first and second molasses to give advantages in boiling, graining and better centrifugal efficiency<sup>31</sup>; the relationship of low grade centrifugal capacity between the purities of sugar and magma<sup>32</sup>; Swedish practice using high efficiency machines<sup>33</sup>; the continuous centrifuging of massecuites using an experimental machine<sup>34</sup>; purification of sugar juice<sup>35</sup>; and the separation of aconitic acid from molasses.<sup>36</sup>

#### Brewing and fermentation industries

A very interesting account by Dolton summarises the application of various types of centrifugal clarifiers used in present day brewing practice.<sup>37</sup> The operation of hermetic clarifiers as used on carbonated beer is described and the advantages of centrifugal clarification and the relative merits of centrifuging as against filtration are discussed. The operation of large sludge space clarifiers and their uses for clarifying hot wort, returns and bottom settlings, and after fining are outlined. Brief reference is also made to continuous sludge discharging machines and their applications. Further information and views on the same subject are given.<sup>38, 39</sup>

Another paper (in Swedish) dealing with the influence of dregs (trub) in wort for top fermented beer, states that centrifugal removal of the dregs appreciably shortens the time for fermentation.<sup>40</sup> Other references to the centrifugal clarification of wort have been made.<sup>41, 42</sup>

In a review of modern trends in distillery practice by McFarlane,<sup>43</sup> information is given regarding processes involving centrifugal separation. The pretreatment of molasses shows that advantages are obtained (a) by chemical treatment and



(Photo: Hopkins Ltd.)

Stationary centrifugal purifier available in different sizes, with bowls rotating at about 10,000 r.p.m. in the case of the smallest machine and at about 5,000 r.p.m. in the larger units. The bowl is adaptable for performing three methods of purification: (1) with positive washing action, enabling microscopic and hygroscopic fibres to be removed from insulating oil, and water-soluble acids, carbon, sludge and other finer impurities from lubricating oils; (2) without positive washing action when contact with water is undesirable; and (3) for clarification when removing only solid impurities and traces of moisture from oils, varnishes, non-inflammable solvents, etc. Features of the machine include positive-drive from motor, enclosed gears lubricated by oil mist, integral drip-proof motor and ball bearings to take thrust and radial loads. All parts of the bowl are protected against corrosion.

subsequent centrifugal clarification of the settled sediment in the case of alcohol production, and (b) by the double centrifugal treatment of the whole of the mash in the case of heavy rum. Processes involving continuous fermentation and re-use of yeast utilise centrifuges for separating yeast from the fermented wash prior to distillation. An additional reference to the latter has been made.<sup>44</sup>

A suggested use of centrifugal clarification of fermented mash would arise if large-scale production of 2,3-butyleneglycol from blackstrap molasses was commenced; centrifugal liquid-liquid extraction of the product would also be a possibility.<sup>45</sup> A patent covering a centrifuge for the clarification of wines and fruit juices has been lodged.<sup>46</sup>

#### Vegetable and fish oils

Some significant developments in the centrifugal processing of vegetable oils have been reported. A new approach has been made to the extraction of oilseeds, particularly those having a high oil content.<sup>47</sup> A system has been designed which

consists of three units in series, each containing a slurry mixer and a Bird continuous separator. The ground seed and fresh solvent are fed into opposite ends of the system bringing about countercurrent extraction. Each unit extracts 90% of the oil input to that unit, and a 20% miscella practically clear and free from fines is discharged from the first unit, while the meal from the third unit contains only a few tenths per cent. of oil.

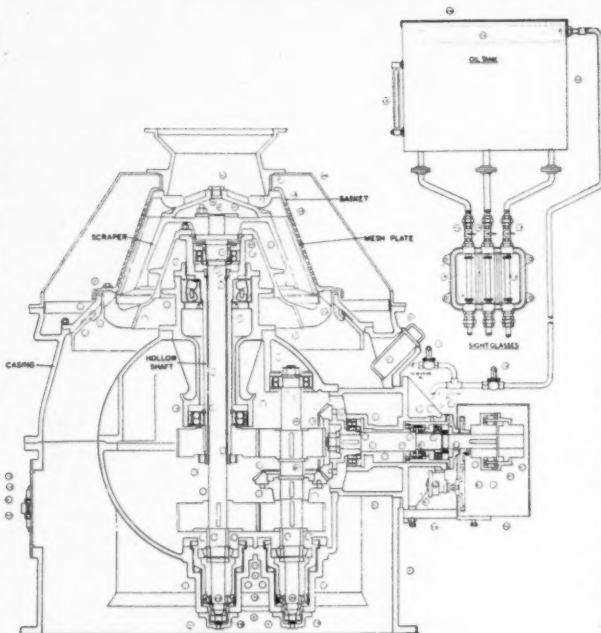
In the conventional extraction process the presence of fines in miscella is a problem. The successful clarification of miscella in two commercial installations using De Laval and Sharples continuous solid discharging machines has been reported.<sup>48</sup> This involves injecting water containing a wetting agent into the miscella so as to water-wet the fines prior to separation. In the case of the De Laval machine the solids and water are discharged continuously and part of this is recycled back through the nozzles so that the phases in the bowl are maintained in equilibrium. With the Sharples machine the solids and water are discharged intermittently through automatically-operated valves which open when the sludge has reached a predetermined depth in the bowl. The merits of the two systems are not compared but typical operating data for the two systems are given.

Other references to the centrifugal production of oils and fats include the dry extraction of palm oil<sup>49</sup>; recovering oil from liquor obtained by working ground soya beans with 0.3% sodium sulphite solution,<sup>50</sup> and fat from glue liquor.<sup>51</sup> As a possible source of fat in the future, the growth of fat-producing yeasts using molasses as raw material is envisaged. The various aspects of the problem including the type of plant required for production

has been detailed and reference is made to centrifugal separators for removing sludge from the raw materials.<sup>52</sup>

Although no new developments in vegetable oil refining have been announced, there is a very interesting report on an American plant producing edible oils and margarine.<sup>53</sup> A new Clayton soda ash continuous refining plant using De Laval separators has recently superseded a Sharples caustic refining system which has been used for some time. The new plant employs three-stage treatment which includes separators for the initial separation of soap-stock, machines at the re-refine stage where the oil is decolorised with caustic soda, and final wash water separators. In the re-refine stage three-phase separation in the centrifuge bowl is overcome by flushing water through the bottom of the bowl, a technique developed by De Laval and covered by Clayton patents in this process; full details regarding the operation of the plant are given. Another description deals with the refining of drying oils and makes reference to continuous refining and degumming by centrifuges.<sup>54</sup>

In the pilot plant fractionation of cottonseed it was desired to produce a meal free from pigment glands and a concentrate of the latter. A full account has been given<sup>55</sup> which includes many references to the use of a continuous horizontal bowl centrifuge running at various speeds for operations in the process where the continuous separation of solids from a liquid was required. A proposed commercial installation is described which includes two continuous horizontal bowl machines for such a process.<sup>56</sup> Reference to laboratory work on the fractionation of cottonseed pigment glands also mentions the use of centrifuges to bring about the separation.<sup>57</sup>



Rheineveld vertical continuous-type centrifuge for dewatering fine coals comprising essentially an outer casing housing the internal rotating parts consisting of a solid vertical centre shaft to which is attached the inner cycloidal scraper, and a hollow shaft (in which the solid shaft rotates) supporting the perforated dewatering basket. Between the basket and scraper is a conical annular space through which the coal travels after being fed into the top mouthpiece and evenly distributed by the distributor cover just below the mouthpiece. Extracted water flows out through ports while the dewatered coal continuously discharges through the base. Nominal capacity of a single machine is 45 tons/hr. input (dry basis) and for this duty it would be driven by a 40-h.p., 720-r.p.m. motor.

[Diagram: Rheineveld General Construction Ltd.]

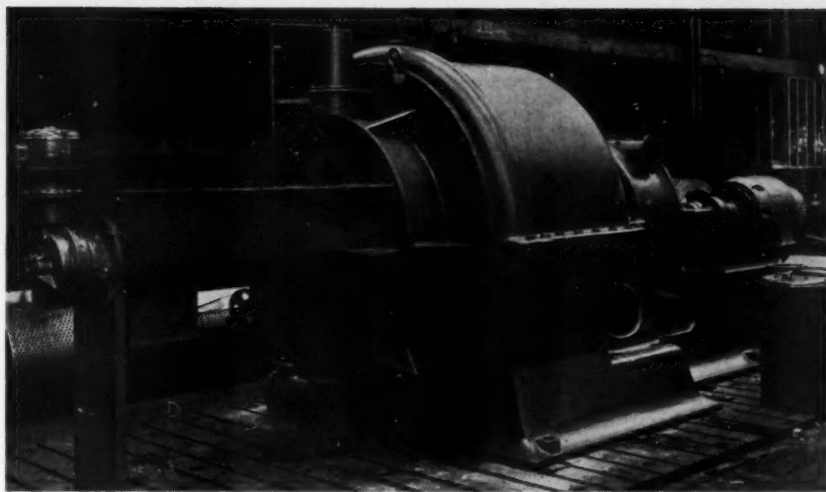
In the treatment of fish oils, passing reference has been made to the conventional process involving cooking, pressing, centrifugal removal of the solids from the press liquor and purification of the oil<sup>58, 59</sup>; the latter reference also mentions the advantages to be gained by the alkali digestion process when dealing with glut landings of herrings. Floating fish factories are assuming greater importance in this industry and several centrifuges have been installed for the processing of fish and fish livers.<sup>60, 61, 62</sup> It is reported that centrifugal recovery of herring oil in Norway is now universal.<sup>63</sup>

A report by Hartman on laboratory and factory tests in New Zealand summarises investigations into the oil and vitamin A recovery from fish livers.<sup>64</sup> Separation of the products obtained by various methods of digestion was carried out using a Sharples centrifuge; of the various methods tried, alkali digestion gave the best results on livers containing more than 30% of oil. This, however, does not appear to be the practice reported in one New Zealand installation<sup>65</sup>; here, high oil content livers are disintegrated, treated with live steam and separated using Sharples centrifuges; no reference to the vitamin A potency is recorded, but with shark livers an oil



[Photo: A. Gallenkamp & Co. Ltd.]

Laboratory centrifuge with head for two 15 ml. and two 50 ml. tubes. The motor is a double impregnated, ball-bearing, universally wound, end-flange mounting type, with a tapered shaft extension. It is fully floating on rubber bushes. The heads are enclosed by a heavy gauge steel bowl through which a continuous stream of air circulates, all parts remaining cool. The exterior surfaces are finished in grey enamel, the bowl being first plated all over to resist corrosion and to present a smooth, easily cleaned internal surface. Various interchangeable heads are available. A hand tachometer can be used for indicating the speed of the machine. All buckets are supplied with moulded rubber cushions.



[Photo: A. & W. Smith & Co. Ltd.]

Rheineveld horizontal centrifuge for separating solids and liquids. The rotating inner drum is fitted with a perforated wall, either with or without a sieving device, and the liquid which is spun out can escape through the perforated wall, e.g. for the extraction of syrup from massecuite. When used as a separator the inner drum is not perforated and the separation is effected by the centrifugal force according to the specific gravities of the liquids and any solids present. A special feature of these machines is that the basket is balanced statically and dynamically, and is mounted on the main shaft permitting high rotation speeds, resulting in faster separations. To resist corrosion, parts which come into contact with the product are of stainless steel and the inside of the main casing is lined with stainless steel. It is also possible to cover such parts with ebonite or lead.

recovery of 70% liver weight has been realised.

The whole subject of continuous processing of fats has recently been presented in book form by Schwitzer<sup>66</sup> in which many references are made to the use of centrifuges. These include details of the De Laval SVK process using nozzle type machines in fish reduction plants, Titan and De Laval separators used for fish liver oil, the Sharples caustic soda process and the more recent De Laval short-mix process for the continuous refining of vegetable oils, and the Sharples process for the production of soap.

#### Dairying

As the first commercial application of centrifuges was introduced into this industry by Dr. De Laval in 1878, it is not surprising to find that centrifuges play an important role in the treatment of milk and associated products. This is reflected in an interesting account of co-operative dairying in Norway which illustrates and describes machines used for separating and clarifying milk, each of which has a capacity of over 1,000 g.p.h.<sup>67</sup> Various methods for continuously producing butter also offer scope for the centrifuge, usually for the initial milk separation to give cream containing 30% fat, followed by reparation to give a concentrated cream of 82% fat, using hermetic machines. A useful description of the different processes with views on the results achieved has been given by King<sup>68</sup>; it is stated that butter so produced has a quality superior to that of ordinary butter and increased yields are obtained at reduced cost. Some such processes have also been patented

recently,<sup>69, 70, 71</sup> and a further patent covers the centrifuging of sour cream products.<sup>72</sup>

#### Lubricating and fuel oils

Due to the exacting requirements of modern engineering installations, it is quite possible that synthetic lubricants will assume greater importance in the future. A review of German war-time methods<sup>73</sup> gives details of their preparation in which three instances of centrifugal separation are involved: (a) Aluminium chloride sludge from an ethylene polymer, (b) an aqueous phase from a higher-olefine polymer, and (c) aluminium chloride sludge, water and cutting oil. No further developments with lubricating and cutting oils have been recorded, but the types of machines in use and their operation have been reviewed.<sup>74, 75</sup>

The continued successful use of centrifugally-treated heavy fuel oil in marine diesel engines is reported; these include trials with a three-cylinder Doxford engine using oil having a viscosity of 3,500 secs. Redwood 1<sup>76</sup>; a typical installation and details of operation for the m.s. *Chilka*<sup>77</sup>; a review of recent progress and developments<sup>78</sup>; purification of the oil in relation to cylinder-liner wear<sup>79</sup> and a report on the performance of the m.s. *Lampania*.<sup>80</sup> Reference is also made to the inclusion of heavy fuel oil separators for a marine engine test bed.<sup>81</sup>

#### Chemical industries

The main types of machines used in this sphere are the continuous horizontal solid bowl type for the separation of solids from a liquid and basket machines for

washing and drying crystals, etc. Among the recent references are: A continuous solid bowl machine for the dewatering of sulphur obtained by the froth flotation of low grade ores<sup>82</sup>; a Bird machine for the removal of salt from caustic liquors used in the manufacture of ethyl cellulose, and a machine for the removal of wash water from the product<sup>83</sup>; a Haubold enclosed horizontal basket machine for the drying and solvent washing of pyrocatechol<sup>84</sup>; a threefold use of a centre-slung basket centrifuge in the production of phloroglucinol<sup>85</sup>; a pilot plant using basket and continuous horizontal basket machines for respectively washing and recovering diammonium phosphate crystals<sup>86</sup>; two continuous solid bowl machines for removing sodium sulphate from an aqueous solution of sodium benzenesulphonate, a similar machine for removing liquors from the separated sodium sulphate after washing, automatic horizontal basket machines for separating and washing sodium sulphite from sodium phenate solution, and a basket machine for the recovery of sodium sulphite from concentrated liquors, all of which occur in the production of phenol by sulphonation<sup>87</sup>; bottom discharge basket, and continuous discharge machines for dewatering and washing polyvinyl chloride<sup>88</sup>; two continuous machines for drying ammonium sulphate crystals<sup>89</sup>; two Bird continuous cylindrical centrifuges for separating and washing potassium chloride at different stages in its production, and a machine for the separation of potassium sulphate crystals,<sup>90, 91</sup> and basket ploughing and Bird continuous machines for the separation of copperas in the processing of titanium pigments, the latter machine also being used for the classification of the calcined product.<sup>92</sup>

#### Miscellaneous applications

These include the use of refrigerated machines for the separation of precipitated protein in the fractionation of human blood plasma<sup>93</sup>; a machine for the separation of materials from blood and plasma<sup>94</sup>; a 16 in. hydro extractor for laundry work<sup>95</sup>; a short description of the use of basket machines for drying crystals and granules<sup>96</sup>; and the use of centrifuges for the recovery of wool grease from wool scouring liquors.<sup>97</sup>

Patents covering processes involving centrifugal separation are as follows: Starch tailings,<sup>98</sup> washing paper pulp,<sup>99</sup> latex concentration,<sup>100</sup> and oil from steam.<sup>101</sup>

Finally reference must be made to a report on the behaviour of Alfa-Laval separators running during a violent earthquake.<sup>102</sup> This should dispel any doubt regarding the safety of such machines when operating under extremely adverse conditions.

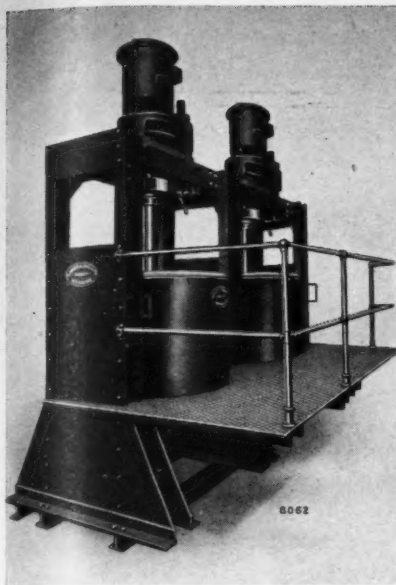
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A high speed centrifuge with a capacity of 14 gal., enabling the machine to run for long periods without interruption. Maintenance of clarification efficiency by preventing accumulation of solids is claimed to be obtained by employing a new design of bowl which is forged in one piece alloy steel and has an internal diameter of 21 in. There is an adjustable pressure discharge device for delivering against back pressures and to long distances, as well as a foamless discharge arrangement. The unit is hermetically sealed against contact with the atmosphere and there is no loss of carbon dioxide. This type of centrifuge can be supplied as a portable or stationary unit, with fast and loose pulleys for belt drive or for V-rope drive and, if desired, variable-speed arrangement. The unit is suitable for handling spirits, fruit juices, essences, paint, varnishes, food preparations and many other products.

[Photo: Lang-London Ltd.]





[Photo: Manlove, Alliott & Co. Ltd.]

Pair of electrically driven centrifugals arranged for bottom discharge

# ION EXCHANGE

## Fundamentals, materials separation, sugar and other food processing

By B. A. J. Lister, M.Sc., Ph.D.

**D**URING the past 12 months important progress has been made towards our understanding of the principles involved in the uptake of ions from solution by synthetic ion exchange materials. The exchanger is considered by Gregor<sup>1</sup> to consist of a number of static anionic groups (*e.g.* sulphonic acid groups) held to a hydrocarbon skeleton which is porous enough to allow an aqueous phase to exist inside. Free water, cations and anions are able to pass through the resin structure. An osmotic process occurs and the exchanger may expand or contract due to differences in its water or electrolyte content. The osmotic pressure of the solution inside the resin is assumed to be opposed by the elasticity of the hydrocarbon structure. Glueckauf and Duncan,<sup>2</sup> using the same model as Gregor, but extending the thermodynamic treatment have more recently derived relationships by assuming that the exchanger phase obeys the laws of strong aqueous electrolytes. The theory satisfactorily explains the experimental results with single electrolytes. Although such treatment brings us nearer to an explanation of the mass of experimental data, a great deal of work is still needed before a completely satisfactory explanation of the process is reached.

Industrial application has been generally along the lines described in the previous reviews in this journal.<sup>3</sup>

### Resin membranes

Research has been in progress in this country and in the United States on the preparation and use of ion exchange resins in membrane form. They offer a very convenient method for studying such properties as resin conductance under varying conditions and can be expected to be of notable assistance in the fundamental studies.

### Separation of materials

Work on rare earth separation has continued. The now well-established citrate complexing elution method has been employed for the separation on a cation exchange resin of europium, gadolinium and terbium,<sup>4</sup> of dysprosium and yttrium,<sup>5</sup> and of europium and samarium.<sup>6</sup> In addition, the separation of the transuranic elements americium, curium and berkelium has been achieved using this technique.<sup>7</sup> The first two of these elements have also been separated from the

rare earths by elution with strong hydrochloric acid.<sup>8</sup> A new eluting agent, ethylene diamine tetra-acetic acid has effected the separation on an anion exchange resin of yttrium and europium.<sup>9</sup>

A complete resolution of a mixture of the alkali metal ions<sup>10</sup> is claimed by elution from a cation exchange column with varying strength hydrochloric acid. Some separation of lithium isotopes by ion exchange is reported by Glueckauf<sup>11</sup> *et al.* and by Gross.<sup>12</sup> The author<sup>13</sup> has developed a method for the complete separation of zirconium and hafnium by elution from a column of cation exchange material with sulphuric acid, use being made of the complex solution equilibria involved.

As mentioned in an earlier review,<sup>3</sup> an important difficulty in the way of industrial application of ion exchange methods for separation is the unsuitability of the technique for continuous operation. This problem is now being examined by Hiester<sup>14</sup> and a continuous separation of lithium and potassium ion using a moving bed technique is being attempted.

The separation of amino acids on anion exchange resins is still receiving considerable attention,<sup>15</sup> and the separation of nucleic acids has been examined.<sup>16</sup> Electromigration of ions in the exchange positions on a resin may offer a further technique for separation purposes.<sup>17</sup>

### Analytical applications

Several reviews<sup>18</sup> have appeared during the last few months dealing with the use of cation and anion exchange resins in analytical chemistry. In addition, reference may be made to the removal of a small amount of titanium from iron by use of complex cyanide formation<sup>19</sup> and to the separation of the anions of the seventh group of the Periodic system.<sup>20</sup>

### Sugar processing

Wide interest is still being taken in the application of ion exchange resins to the sugar industry, and several reviews have appeared.<sup>21</sup> The decolorising ability of the resins is claimed to be outstanding. One difficulty found is that, after ion exchange treatment, raw juice is opaque and difficult to filter; this, however, can be overcome by coagulating at the optimum pH, the addition of a filter aid, or a combination of the two.<sup>22</sup> The best results seem to be bound up with good preclarification methods.<sup>23</sup> Jacobs and Cole<sup>24</sup> have found that, after demineralisation, the slightly

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coloured sugar easily turns brown due to melanoidin formation, but this can be prevented by the addition of sulphites. Favourable effects on the bacteriological condition of treated sugar are dealt with by Porter.<sup>25</sup>

Two further aspects of use in the sugar industry may be mentioned. The recovery of glutamic acid in the demineralisation treatment of beet sugar is described in an American patent,<sup>26</sup> and Smit<sup>27</sup> has studied the inversion of sugar juices with cation exchange agents in the hydrogen cycle. He claims that the process yields products with higher purity than those obtained by other methods and that it is suitable for continuous operation.

### Other food industries

The effect of cationic and anionic exchangers on milk, whey and ice cream mixture has been examined<sup>28</sup> and it has been shown that evaporated milk is stabilised against sterilisation. Dried cream is more soluble and does not oil off on reconstitution when properly treated with cation resins before drying. For the best results the calcium ratio must be reduced from 1.2 to below 0.75 in the cream. The use of cationic exchange materials permits the manufacturing of stabilised high-solids evaporated milk with the addition of less sodium phosphate. A study has been made of acid removal from wines with anion exchange resins.<sup>29</sup>

To summarise, although considerable work in the field of ion exchange has been reported, and progress has been made in industries where the use of resinous exchangers is already established, no important new industrial applications have come to light during the past year. In Britain as well as in the United States the overwhelming proportion of ion exchange materials produced is still used in water treatment plants.

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## Equipment for New Oil Refineries in Bolivia

UNTIL recently, Bolivia's oil-refining facilities comprised mainly a 1,000 barrels/day shell still located in the Camiri producing field. Very recently two new refineries and a 300-mile pipeline system connecting them with Camiri have been added which will shortly bring Bolivia's oil-refining capacity up to about 11,000 b.bl./day, sufficient to meet domestic requirements for the major petroleum products.

The first and smaller refinery, located at Sucre, went into operation last October. Its 1,000 b.bl./day unit is said to have been run up to about 300 b.bl./day above its design capacity. Work is now under way to add a 3,000 b.bl./day unit. In general, facilities are much the same as at Cochabamba, which is the second and largest of the two refineries.

### The Cochabamba plant

The Cochabamba refinery has a capacity of 5,000 b.bl./day. It includes a light-ends aviation petrol unit consisting of three main towers, two 60-tray and one 70-tray, together with heat exchangers, accumulators and reboilers.

The Camiri crude which will be processed is 64°F. A.P.I. gravity, amber in colour and yielding about 65% petrol. For the maximum high-octane aviation petrol production from operation of the light-ends unit, about 180 b.bl./day of alkylate will need to be imported. This alkylate, along with lubricating oils and special products which the refineries do not produce, are expected to be about all of Bolivia's future petroleum imports.

The topping unit at Cochabamba of 5,000 b.bl./day rated capacity includes furnace, fractionating column, strippers and heat exchange. Ethyl facilities provide for the injection of 4 c.c./gal.

The boiler house with two Foster Wheeler 40,000 lb./hr. boilers supplies process steam and runs two 300-kW turbo-generators. Water supply comes from a nearby river, and is treated in a complete Permutit settling, flocculation and softening system. The oil separation consists of

two separation bays with skimmers and sloop pump-out. The refinery also has stabilisation equipment and caustic-wash facilities. The transfer pump house consists of 10 steam 150 gal./min. pumps.

Tankage at Cochabamba includes one 80,000-b.bl. crude-storage tank, approximately 75,000 b.bl. total storage for products, and other refinery operating tankage. About 60 men will be required to operate the completed plant. Auxiliary facilities include living quarters, cafeteria, products-testing laboratory, machine shop and warehouse, and loading rack and barrelling shed.

At both refineries a new liquid fire-protection system has been installed. This injects *Aerofoam* liquid into the fire-water main to obtain a 10% solution fed directly to the *Aerofoam* chambers on storage tanks.

The oil pipeline is claimed to be the highest in the world, rising in places to as much as 11,000 ft. The system consists of 255 miles of 6-in. pipe from Camiri to Cochabamba and a 45-mile 4-in. lateral branching off near near Zudanez to Sucre.

The two refineries were designed and built by the Foster Wheeler Corporation and all equipment was brought from the U.S. Estimated cost of the two projects, including the extension at Sucre, was \$11,700,000.

### Centrifuging

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# Production and Uses of Gallium Metal

Although as plentiful as lead, gallium is so widely distributed that its recovery is difficult and output is measured in grams. The metal has unique properties; for instance, it has one of the longest liquid ranges of any element, a characteristic which has suggested its use as a heat exchange medium. It is also used in organic syntheses and for the manufacture of low melting point alloys. Here are some notes on the occurrence, recovery, properties and applications of this unusual metal.

GALLIUM, though only recently publicised, is not a newly discovered element. In 1861, Mendeleeff presented his famous paper 'The Relation of the Properties to the Atomic Weights of the Elements.' Subsequently, he predicted properties of a number of undiscovered elements. Three he designated ekasilicon, ekaboron and ekaaluminum. These later became known as germanium, scandium, and gallium, respectively.

Gallium was the first of the three eka elements to be identified and isolated. This was accomplished in 1875 by the French chemist Lecoq de Boisbaudran. This was the culmination of a brilliant 15-year study of spectra.

## Distribution

As might be expected from its proximity to aluminum, gallium is widely distributed in the earth's crust. It exists in roughly the same quantity as lead, about 15 gm. per ton of earth. Lead, however, is concentrated at favourable spots while gallium is almost universally disseminated but nowhere in significant commercial concentration. As far as is known, there is no mineral that contains gallium as a major constituent—the nearest exception being germanite, a complex zinc-copper-arsenic-germanium sulphide found in the copper ores of Mansfeld, Germany, and in the Tsumeb copper-lead mine, South West Africa. This mineral usually contains from 0.1 to 0.8% gallium with one specimen reported to have contained as high as 1.85%.

Gallium is frequently associated with germanium in the comparatively rare mineral germanite and in coals. Unfortunately germanium is rare. Further, almost any sample of earth or clay, of which, of course, aluminium is a major constituent, will show a gallium content on the spectrograph often in the order of 50 gm./ton or considerably over the average for the earth's crust. It is also found in bauxite, the source of commercial aluminium, and accumulates in the circulating liquors of the Bayer process from which it may be recovered.

## Production

Gallium is closely tied to the Tri-State lead and zinc field of South West Missouri, South East Kansas, and North East Oklahoma. The story of gallium in the Tri-State district began in 1915 with the early work of F. G. McCutcheon, now manager of the Eagle-Picher Zinc Smelter at Henry-

etta, Oklahoma. The leady heels or residuum left in the retorts was cast in slabs and stacked outdoors pending shipment to a lead smelter. Following rain, McCutcheon noticed beads or droplets of metal exuding from the slabs. On analyses these beads proved to be an alloy of about 94% gallium and 6% indium. The only explanation for the formation of the beads of metal was that the heels were a complex mixture of metals, including sodium and calcium. The latter are believed to have reacted with the rain water to release the beads of rare metal alloy. McCutcheon subjected several slabs to steam and reproduced the phenomena, collecting 300 gm. of alloy from one 700 lb. lot of heels so treated.

He developed recovery processes and produced the world's first pound of gallium metal, and by 1944 had recovered some 6,000 gm. of the metal.

The Eagle-Picher Research group at Joplin meanwhile had become interested in gallium recovery. The Eagle-Picher lithopone plant at Argo, Illinois, was processing 10 tons/day of roasted zinc ore and producing about 1,000 lb./day of iron mud lead residue. McCutcheon pooled his knowledge with that of the research department and a process was developed for handling this material. It consisted of a

caustic leach that dissolved aluminium and gallium compounds and some silica; precipitation as hydroxides; filtration and dehydration of the cake; hydrochloric acid leach; precipitation as hydroxides; solution of cake in hydrochloric acid; ether partition; electrolysis in caustic electrolyte; and fractional recrystallisation of the recovered metal. Since only 0.07% gallium is concentrated in the iron mud leach residue, associated with about 10% aluminium and 15% iron as well as variable amounts of a great many other elements, the difficulty and expense of the separation can easily be realised. Gallium costs \$2.50 to \$7.50 gm. depending on the quantity purchased. A new recovery method must be developed for each new gallium raw material. Furthermore, use of strong hydrochloric acid is almost an essential step of any recovery process, introducing a severe materials-of-construction problem, especially on a pilot plant scale.

## Chemistry

The chemistry of gallium is comparable to that of aluminium and also bears a marked resemblance to that of the two succeeding elements, indium and thallium. The stability of the compounds of these

TABLE I. PHYSICAL PROPERTIES OF GALLIUM

Atomic No. . . . .	31
Atomic weight . . . . .	69.72
Isotope abundance—Mass No. 69, Pct. . . . .	61.2
Mass No. 71, Pct. . . . .	38.8
Crystal structure . . . . .	Orthorhombic
Density—20° C. (s.), gm. per cc. . . . .	5.907
29.65° C. (s.), gm. per cc. . . . .	5.9037
29.8° C. (l.), gm. per cc. . . . .	6.0948
Specific volume at melting point (solid) . . . . .	0.1694
(liquid) . . . . .	0.1641
Melting point . . . . .	(85.5° F.) 29.75° C.
Boiling point . . . . .	(3,601° F.) 1,983° C.
Latent heat of fusion—gm. cal. per gm. . . . .	19.16
Btu. per lb. . . . .	34.5
Latent heat of vaporisation, gm. cal. per gm. . . . .	1,014
Heat of combustion (2Ga + 3O = Ga <sub>2</sub> O <sub>3</sub> ), k.cal. . . . .	259
Vapour pressure—1,315° C. (2,399° F.), mm. Hg . . . . .	1.0
1,726° C. (3,139° F.), mm. Hg . . . . .	100
1,983° C. (3,601° F.), mm. Hg . . . . .	760
Specific heat—solid, 16° to 24.2° C., gm. cal. per gm. . . . .	0.0926
liquid, 21° to 130° C., gm. cal. per gm. . . . .	0.0977
Linear thermal coefficient of expansion—0° to 30° C., cm. per cm. . . . .	1.8 x 10
Volume resistivity at 20° C., microhm/cm. . . . .	56.8
Volume conductivity at 20° C. (copper/100 Pct.), Pct. . . . .	3.04
Volume resistivity of liquid at 46.1° C., microhm/cm. . . . .	28.4
Magnetic susceptibility at 18° C., cgs. units . . . . .	0.24 x 10
Reflectivity—4,360 Å, Pct. . . . .	75.6
5,890 Å, Pct. . . . .	71.3
Electrochemical equivalents—mg. per coulomb . . . . .	0.24083
gm. per amp. hr. . . . .	0.86698
lb. per 1,000 amp. hr. . . . .	1.91137
Viscosity—77° C., Dyne-sec. per sq.cm. . . . .	0.01612
1,100° C., Dyne-sec. per sq.cm. . . . .	0.00578

four elements decreases with increasing atomic weight. As a corollary, the ease of reduction to the metal increases with the atomic weight. All are permanent in air at ordinary temperatures, but when heated develop a protective oxide coating. The normal hydroxides are amphoteric in the case of the first three elements and basic only in the case of thallium. Like aluminium, gallium forms alums.

The usual valence is three. With increasing atomic weight, the tendency to form compounds of lower valence becomes more marked, this property being most noticeable in the case of thallium. Aluminium normally shows no valence other than three while gallium forms a number of divalent and even some monovalent compounds.

### Properties

Gallium metal is most unusual in its properties. It has one of the longest liquid ranges of any element, melting at 29.75°C. (85.5°F.) (about 70°C. higher than mercury) and boiling at 1983°C. (3601°F.). There is some difference of opinion on the boiling point, values up to 2100°C. and higher being reported. The boiling points of its congeners, aluminium and indium, are of the same order, while that of thallium is about 500°C. lower. The vapour pressure of gallium remains quite low at comparatively high temperatures, being only 1 mm. at 1315°C. (2399°F.).

Gallium is one of the few metallic elements that expands on solidifying. The liquid density at 29.8°C. is 6.095 gm./c.c., while the solid density at 20°C. is 5.907 gm./c.c., roughly a 3% expansion on solidification—almost as great as that of bismuth, 3.3%. If packed directly in a glass container as the liquid metal, expansion on solidification breaks the container. On warming a little, the gallium melts, and flowing like mercury, is often lost or contaminated. One supplier packages the liquid metal in rubber bulbs. Eagle-Picher supplies the metal as clean crystals dipped from the melt while cooling, packed in sealed *Pliofilm* bags. Should the metal accidentally become warm and melt it cannot escape. On resolidifying, the elastic *Pliofilm* prevents rupture and loss.

Gallium crystallises in the orthorhombic system, and beautiful specimens are produced. The exact shape of the crystals varies from thin square plates to thick and massive spear points.

As might be expected from its position in the periodic table, gallium is a chemically active metal. The literature contains an imposing list of alloys and solid solutions with other metals. Since certain suggested uses for gallium are based upon its long liquid range and low vapour pressure at elevated temperatures, the matter of reactivity and of a suitable container is important.

Important physical properties of gallium are summarised in Table I.

### Uses

A wide variety of uses has been suggested for gallium, ranging from low melting-point alloys to the treatment of bone cancer. The first use proposed was as fill material in high temperature thermometers. Practical difficulties arose such as the lack of availability of quartz glass tubing in the required bores and o.d. sizes. It is understood that some thermometers have been produced, but the cost has been very high and the results far from satisfactory.

The use of gallium as a backing material for optical mirrors has been suggested as it reflects a high percentage of the incident light. For certain purposes in atomic and astrophysical spectrum research work the gallium lamp has proved quite useful. The Bureau of Standards reported satisfactory service from quartz vacuum lamps using Ga-Zn and Ga-Cd mixtures to replace mercury.

### Heat exchange

The Atomic Energy Commission has investigated gallium as a possible heat-exchange medium. The favourable thermal characteristics, particularly the long liquid range and low vapour pressure, encourage consideration of its use in extracting heat from a high level, since any atomic energy power plant must use temperatures considerably above those of conventional steam-power plants if even a small part of the potential energy is to be utilised. However, gallium's considerable reactivity as expressed in terms of attack on possible container materials thus far has delayed its application in this field.

The addition of a small percentage of gallium oxide to relatively high grade uranium-base material in the form of the oxide, and subsequent fractional distillation in the d.c. arc, has made it possible to determine 33 volatile impurity elements at concentrations as low as a fraction of a part per million.

### Organic synthesis

One principal use of gallium reported by the Germans is in organic synthesis. Its chloride salts act as catalysts in the Friedel-Crafts reaction. In some cases a smaller amount of catalyst is required than when other chloride salts are used; in other cases a higher yield is obtained or the reaction time is shortened.

### Alloys

Low-melting point alloys are formed by gallium with indium, tin, and aluminium, and may find use in fire alarm systems. The addition of 2 to 4% gallium improves the mechanical properties of aluminium. Gallium increases the hardness of aluminium ternary alloys. An iron alloy containing 3% gallium and 14% nickel resembles beryllium and titanium steels in hardenability. The addition of 4.5% gallium will harden magnesium on heat treating. Gallium also may find a place in electrical contact alloys.

Before 1932 gallium was available only in small quantities for laboratory research purposes. In 1932 the Chemical Manufacturers' Association of Leopoldshall (Germany) began to recover gallium from the residue from the Mansfield copper schists. By 1937, the annual production had risen to about 50 kgm. Directly prior to World War II, German production was about 300 lb. p.a.

From 1943 to 1945, the Anaconda Copper Mining Co., in connection with its recovery of indium, produced several thousand gm. of gallium. In 1946, the Eagle-Picher Co. was the only producer. In 1947, Aluminium Co. of America entered the field and in 1948 the Saratoga Laboratories, Inc., started production. Since 1946, a licence has been required for the export of the metal from the U.S.A.

(A. P. Thompson and H. R. Harner, *Journal of Metals*, February 1951)

## Recent publications

**Industrial fans.** Steel plate fans are manufactured in many types by Sturtevant Engineering Co. Ltd., but can be divided into three main classes, the fundamental variation being in the form of the impeller blade which may be radial, forward curved, or backward curved in relation to the direction of rotation. These three forms are basic, each having its own particular sphere of service, but various constructional modifications are made to meet the demands of modern fan engineering. Publication 2203 issued by the manufacturers indicates the main types in this extensive range.

One example shows a rubber-lined steel plate paddle blade fan exhausting acid fumes from a rotary drier forming part of a fertiliser granulating plant. Another model is designed for heavy duties such as the collection of wood refuse, conveying of chips, wood pulp and other bulky materials. It will also handle stringy materials without difficulty. Other uses covered by the range include air conditioning, mechanical draught, cooling internal combustion engines, supplying air to oil-burning installations, and fume and dust exhaustion.

**Rust protection.** Iron and steel surfaces pre-treated with *Jenolite* rust preventive and then finished by painting, lacquering, enamelling, etc., as required, are claimed to be in every way as resistant as galvanised and tinned surfaces. Details of this method of rust protection are given in *Jenolite News*.

### 'I.C.E.' September

**MIXING** will be the main theme of this issue.

# The Electron Microscope and its Uses

By J. Rose, B.Sc.

The electron microscope is as great an advance upon the optical microscope as was this instrument upon the naked eye. By its use man's vision has been improved by more than 200 diameters compared with the highest resolutions previously possible. In this concise article, the author explains this important research tool and describes its uses in the chemical process industries and in metallurgical and biological studies.

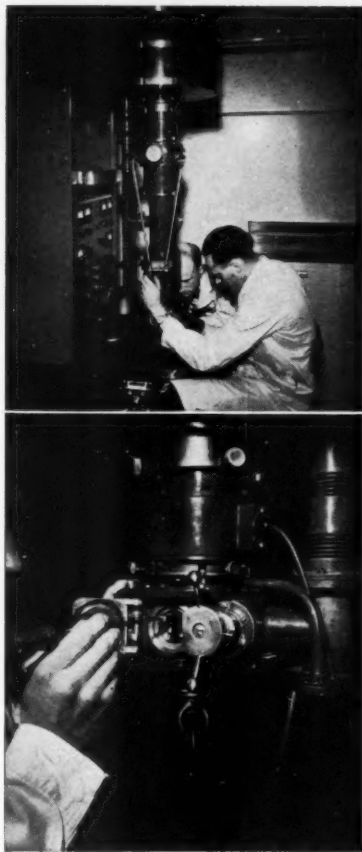
THE majority of solid and solid-liquid systems are heterogeneous, the degree of heterogeneity varying from that found in simple catalysts to that of highly complex biological structures. The major difficulty in studying such systems lies in the circumstance that the heterogeneities often go beyond the limits of visual resolution. The compound light microscope cannot resolve beyond approximately  $2 \times 10^{-4}$  mm. owing to the wavelength of the illumination; the ultra-violet microscope, using light of shorter wavelength, extends this resolving power by a factor of two.

Although much of our present knowledge of biology and medicine, of metallurgy, and even of physics and chemistry, is derived from microscopic observations, yet in all these sciences there are unsolved problems which relate to structures which are beyond the resolving power of optical instruments. The electron microscope fills the gap; it is capable of resolving structures down to  $10^{-6}$  mm., i.e. to molecular dimensions; this increase in resolution is due to the very short wavelengths associated with electrons (0.05 Å at 60,000 volts). The electron instrument brought about as big an increase of resolving power (of about 200×) as its predecessor, the optical microscope, did compared to the naked eye. The electron microscope covers the range of dimensions between its optical counterpart on the one hand and X-ray equipment methods on the other. Its lower limit of resolution,  $10^{-6}$  mm., is due chiefly to asymmetry of objective lens, pole pieces and spherical aberration. Almost certainly its range will be considerably extended in the next few years.

## The instrument

The most generally useful of the various types of electron microscope is the transmission instrument, intended solely for the examination of thin specimens by transmitted electrons. The structure resembles that of the light microscope; the electron beam replaces the light beam and the 'electron' lenses, i.e. magnetic or electrostatic fields, replace the corresponding light lenses for focusing the electrons. Owing to the peculiar properties of an electron beam, the electron microscope must operate under vacuum conditions.

This instrument also differs from its optical counterpart in its greater depth of focus, which results in many planes of



[Photo: Leonard Hill, London]

One of the electron microscopes used for the study of viruses at the Mill Hill, London, laboratories of the National Institute for Medical Research. This R.C.A. instrument allows a final magnification on photographic paper of up to 150,000 diameters. Insertion of a specimen into the microscope is shown in the bottom photograph.

the image coming into focus together; the images are thus superimposed one upon another, but nevertheless a clear idea of the three-dimensional structure of the specimen can often be obtained by stereographic work.

Whereas the 'light' microscope 'sees' in terms of the opacity of the specimen, the electron microscope 'sees' in terms of the scattering power of the specimen for electrons.

## The range covered

Some overlapping may occur between

the uses of certain types of light microscopes and their electronic counterpart. There is, for instance, a considerable field of application for the electron microscope at relatively low magnifications (order of 3,000 ×), owing to the greater definition attained in the pictures. Both instruments share the possibility of studying surfaces by means of replica techniques which involve the examination of an extremely thin and accurate cast of the surface of the specimen. There is, though, a wide range in which the electron microscope has a virtual monopoly; it alone can determine the structure of macromolecules and viruses, for although indications of shape and size of particles in the macromolecular range of dimensions may be obtained by other methods, such as centrifugation, electrophoresis and determination of birefringence, only the electron microscope can record the structure in a picture.

## Shadow casting

This advantage is often amplified by the application of 'shadow-casting,' i.e. by allowing vaporised heavy metal atoms to strike the surface of the specimen obliquely. As thin films of many heavy metals, i.e. uranium, chromium and gold, have no structure except at the highest possible magnifications of the electron microscope, and as they scatter electrons more than films of equal thickness of organic materials, the image contrast is increased and small projections on the surface are brought out in strong relief. The 'shadow-casting' technique gives precise information regarding the heights of surface irregularities.

## Instrumental developments

Orthodox electron microscopy reached a milestone in its development in 1947, when Hillier and Ramberg compensated the astigmatism of their objective lenses and almost realised the theoretical resolving power of the instrument. Further improvements in the latter at present appear to be difficult, owing to errors in construction which are almost unavoidable; thus, for a resolving power of about  $10 \text{ Å}$  the objective must be round to within one part in 10,000; the 'roundness' of the objective must be improved to one part in 1,000,000 in order to increase the resolving power tenfold. It was suggested that higher resolving power might be obtained by means of a proton beam, in-

stead of an electron beam; but so far practical difficulties have precluded its use.

### Diffraction electron microscopy

Gabor<sup>1</sup> hit upon the idea of diffraction microscopy in which the image of the object is obtained in two steps. The first is carried out with electrons; a special kind of diffraction photograph or holograph, unlike the original, is obtained. The likeness is restored in a second process, carried out with visual light, in which the holograph is used as a diffraction medium; a resolving power of a higher order of magnitude is obtained. It is believed that the monochromatisation of the electron beam by a factor of 100 times better than at present would result in considerably greater resolving power. Diffraction microscopy can, however, explore phase contrast to the fullest extent; a superior resolution of thin structures of materials of low atomic weights would also result.<sup>2</sup>

### Direct reflection microscopy

Borries<sup>3</sup> used high-speed electrons, reflected from a metal surface at grazing incidence, for imaging; the micrographs, however, suffered from a strongly foreshortened view of the surface studied, and this constitutes a serious limitation of the method. Despite these difficulties, some interesting observations were made by Raether,<sup>4</sup> who studied the structure of polished surfaces; surface protuberances of about 100 Å could be observed.

Various research workers proposed the use of electron microscopes operating at higher voltages (400,000 to 1,000,000 V) in order to penetrate thicker sheets of metals (about 50,000 Å thick); a 1,000 KV instrument is under construction at the Bureau of Standards, Washington. If million-volt instruments prove successful, the scope of the transmission instrument will be greatly increased.

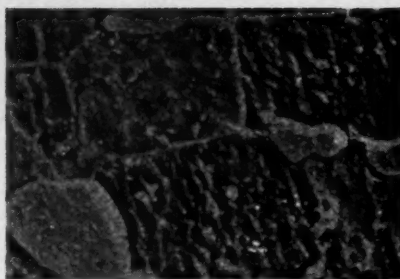
### Emission electron microscopy

The 'emission-type' microscope utilises the electrons emitted by the specimen itself.<sup>5</sup> A resolving power of 200 Å is claimed by Mecklenburg.<sup>6</sup> This general method is subject to the limitation that many metallic samples cannot be heated without undergoing structural changes. Impregnation of the surface with radioactive isotopes<sup>7</sup> is a variant of the 'emission method'; the general complexity of nuclear reactions and the high and variable velocities of emitted electrons introduce grave difficulties.

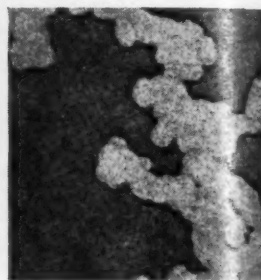
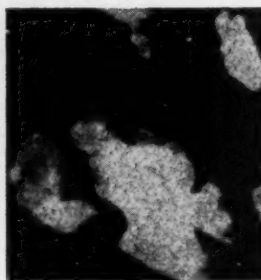
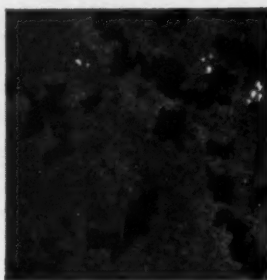
### Electron microanalysis

Carstaing and Guinier<sup>8</sup> estimate the x-rays which are emitted by a surface when bombarded with electrons, by means of Geiger counters; they claim to be able to perform a chemical analysis on an extremely small area of the specimen.

A rather different approach to electron microanalysis is the use of velocity-



Left: Tempered carbon steel ( $\times 20,000$ ). Right: Iron oxide powder ( $\times 18,400$ )



Left: Carbon black shadowed with Pd ( $\times 20,000$ ). Centre: Cornwall clay ( $\times 10,000$ ). Right: Carbon black particles ( $\times 60,000$ ).

analysers; the latter are based on the fact that high-speed electrons suffer a discrete loss when traversing a thin film or a fine powder, and that this loss is characteristic of the material of the specimen. Two different experimental procedures have been proposed to take advantage of this loss.<sup>9, 10</sup>

### Applications

The applications of the electron microscope to the study of problems in chemistry are numerous and may be divided into the four classes discussed below.

#### Determination of particle size

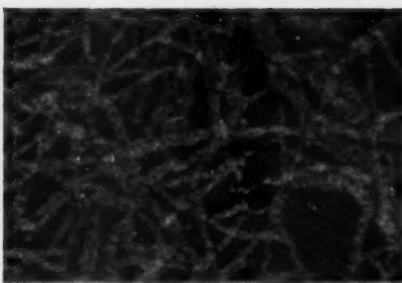
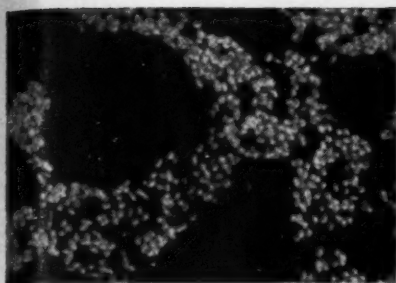
The first is the determination of the particle sizes and shapes of solids. This information is often of value in connection with their manufacture and use; the properties of colloidal systems especially depend greatly on the size and shape of the particles which constitute one of the phases. Various methods of measuring the size of the latter, such as absorption, x-ray scattering, ultra-centrifugation and electrophoresis give as a rule the mean size, but not the particle size distribution, and while techniques such as streaming birefringence and light scattering may give some indication of particle shape, the information they do give is only sketchy. A paper<sup>11</sup> by Turkevitch and Hillier contains information on this subject; it surveys various colloidal systems and discusses morphological forms and their size, shape and distribution.

A wide range of industrially important materials come within this definition, and they include carbon blacks in various commercial products such as printer's ink,<sup>12</sup> rubbers,<sup>13</sup> the colloidal platinum and plati-

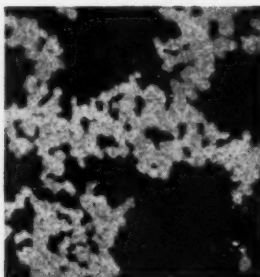
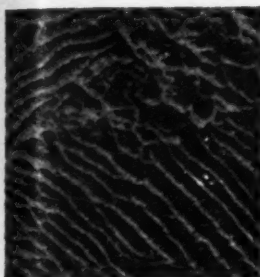
nised asbestos used as catalysts,<sup>14</sup> activated charcoal<sup>15</sup> and alkali soaps<sup>16</sup>; many paints and lacquer pigments such as titania, zirconia, iron oxide and azo blue,<sup>17</sup> as well as dusts and smokes,<sup>18</sup> which have been studied in connection with industrial health problems. The electron microscope appears to be the instrument of choice in this field, as it is generally agreed<sup>17</sup> that particle size may be an important factor in the toxicity of airborne dust, which can cause injury to the lungs; a high proportion of dust consists of particles which cannot be seen in the light microscope, but which can be studied with the electron microscope. Work has also been carried out, in the oil industry, on the particle shape and size of clays (drilling mud and catalyst support<sup>19</sup>), on iron-oxide base catalysts,<sup>14</sup> lubricating oils,<sup>19</sup> and on greases.<sup>15, 18, 20</sup> Metal powders have been studied, as has also the relationship between the properties of certain alloys and fine powders (tungsten carbide cutting tools, etc.<sup>21</sup>).

#### Study of chemical processes

Little actual work has, as yet, been done in this attractive and doubtless fertile field. However, investigations are known to have been started on the action of catalysts, the setting of cement; the age-hardening process in certain alloys;<sup>22</sup> the effect of ageing on the structure and gel properties of vanadium oxide solutions;<sup>23</sup> the metallo-organic precipitates in inorganic analysis;<sup>24</sup> the relationship between crystal habits of substances and their chemical properties;<sup>25</sup> and on the solubility of inorganic materials.<sup>26</sup> Studies have also been made on the photographic developing process;<sup>27</sup> observations on various stages of this pro-



Left: *Bacillus prodigiosus*, optical microscope ( $\times 2,000$ ). Right: Tobacco mosaic ( $\times 50,000$ )



Left: Eutectoid steel, Pearlite ( $\times 10,000$ ). Centre: Quenched and tempered eutectoid aluminium bronze ( $\times 7,500$ ). Right: Tungsten powder ( $\times 10,000$ ).

cess may revolutionise the accepted theories of these phenomena.

#### Studies of 'organised' materials

Studies of the structure of materials, in which there is a degree of organisation, such as diatoms, cellulose, rubber and various fibres, constitute another field of application of electron microscopy in chemistry. Even the unstressed simple physico-chemical phases of a resin, though characteristically structureless under the optical microscope, do exhibit a certain degree of organisation when viewed under the electron microscope.<sup>28</sup>

Inorganic materials, such as iron oxide hydrates, alumina sols and gels, and asbestos<sup>31</sup> have been studied and found to be fibrous in structure. Cellulose (cotton and hemp) fibres have been investigated intensively,<sup>29</sup> and natural and artificial silks<sup>30</sup> have also been studied; an extensive investigation has been made into the structure of wool<sup>34</sup> and of the phenomena of 'felting', 'shrinking' and 'shrink-proofing'.<sup>31</sup> Rubbers have also been examined, and the structure of rubber latex, buna latex<sup>32</sup> and stretched rubber membrane<sup>33</sup> studied with reference to vulcanisation and milling.

Replica studies have been made of dyed nylon.<sup>35</sup> The electron microscope appears to be greatly superior in this field to its optical counterpart, because the crystal size of dyes and pigment which have been applied to textile materials is almost invariably below the limit of the light-microscopic resolution. The size, shape and distribution of the dyestuff particles on the fibre can be investigated; it is also possible to identify a dyestuff using only extremely small samples of the textile material.

#### Biological applications

In the field of biology the submicroscopic dimensions inaccessible to light microscopy are readily explored with the electron instrument. An enormous variety of objects is being subjected to extensive and careful research to ascertain their structural details. This embraces bacteria, viruses, bacteriophages, protoplasmic fibrils, tissue-culture cells; the submicroscopic organisation of the liver, nerve and muscle, chromosomes, chloroplasts; the antibodies; antibiotics and mutation-inducing chemicals and physical agents. Most of the filtrable viruses can be observed through the electron microscope. Isolated virus particles have been shown to form beautiful crystalline arrays in three dimensions. A fascinating study of chromosomes and genes was made by Pease and Baker;<sup>36</sup> the current advances in genetics and cytology of micro-organisms have brought us to the threshold of important discoveries,<sup>37</sup> and the range of useful applications of the electron microscope in the medical sciences is being steadily broadened by the development of new techniques, and an enormous field of fine structures of normal and pathological tissue is becoming accessible for research.

#### Metallurgical applications

It is difficult and sometimes impossible to interpret the properties of metals and alloys by means of x-ray diffraction analytical results. The dimensions of microcrystalline structures range from about 10 Å to several microns. The electron microscope is eminently suitable for use in this range, and it affords an important tool for research in structural metallurgy; it is, however, impossible, at the present time,

to study metals and alloys by direct reflection methods as in optical microscopy, and replica techniques have to be employed.

Although numerous attempts have been made to evolve a 'direct' method of viewing the metal by means of a reflection microscope with high-voltage instruments (1,000,000 V) and with 'emission' microscopes, the transmission-type instrument is still supreme.

Initially the adoption of the electron microscope was less enthusiastic than might have been expected, considering its potentialities. In the opinion of many workers this was due to a confusing lack of reproducibility of results, even in the same hands with the same specimen, but this situation has now been remedied. A symposium<sup>38</sup> on the 'Metallurgical applications of the electron microscope,' held in London in 1949, showed the wide range of topics studied, and it revealed to some extent the relationship between requirements and possibilities in the field of metallurgical applications. Enthusiasm is now greater, and a variety of problems are being subjected to the scrutiny of the electron microscope. Some of the subjects being investigated are fracture surfaces, age hardening, metal powders, structures of light alloys, of brasses, creep in steel, the  $\sigma$  phase in stainless steel and certain isothermal transformations in eutectoid steel. It is hoped also to apply the electron microscope to the investigation of hitherto unsolved problems, such as the irresolvable constituents of hardened and tempered steels; the existence, or otherwise, of 'amorphous' metal on slip planes or at grain boundaries, the formation and growth of transformation nuclei and the relation between structure and strength.

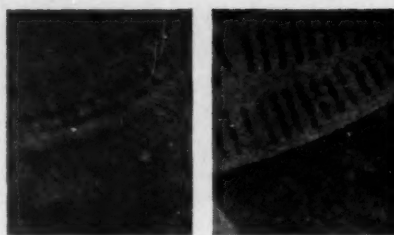
Some remarkable successes have already been achieved; the existence of submicroscopic slip lines has been demonstrated on aluminium crystals;<sup>39</sup> the elongated precipitate characteristic of the  $\theta'$  phase in the hardening of Al-4% Cu alloys was registered,<sup>40</sup> the dependence of structural hardening of light alloys on the deformation of the crystal lattice was also examined.<sup>6</sup> In addition, the high resolving power of the electron microscope has demonstrated the limitations of many of the accepted polishing and etching techniques. The great depth of the field of this new instrument has led to new possibilities in the preparation of metallographic specimens.

In general the development of the electron microscope has improved man's vision by more than 200 diameters. The instrument fills the gap between x-ray methods and optical microscopy and problems of thin films, small particles and surfaces can be studied by means of this powerful tool in a way hitherto impossible.

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Comparative micrographs of diatoms at same magnification ( $\times 2,375$ ). Left: Optical microscope. Right: Electron microscope.

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<sup>47</sup>Various authors: Ref. 1, pp. 113-166.  
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## Revival of West German Synthetic Rubber Industry

THE West German synthetic rubber industry hopes to be producing 20,000 tons of buna a year by the end of 1952. The industry was recently freed from an Allied ban on the manufacture of buna. Before the war, the Reich was a major world producer of this rubber. During the next few months, however, West Germany's buna output will be negligible. War damage, the effects of dismantling, and the shortage of capital and coal will take time to overcome. Capital is urgently needed to rehabilitate plants which have been idle for six years. The industry is not expecting any large inflow of capital, however, as the Government has declared that the basic industries must come first.

The coal shortage hits buna producers particularly hard. The production of buna requires a relatively large quantity of coal compared with other industries—about ten tons of coal for one ton of buna. The production of 20,000 tons of buna a year would therefore require about 200,000 tons of coal and this is not expected to be available for some time, according to the German coal sales organization in Essen. Buna production can at present be resumed

only at the chemical works in Huels (Ruhr). The plant at Ludwigshafen in the French zone, was so badly damaged it will take an estimated two to three years to get it going again. The Bayer works in Leverkusen (Ruhr) will specialise in perbunan, an oil resisting rubber.

The director of the Huels works states that it is planned to take up the development of the primary product, butadiene, and of buna, the finished product, in two stages. In the first stage the butadiene plant would be developed so that it could produce 500 tons of butadiene a month. Most of this would be made at Huels into buna S3, the best type of German synthetic rubber. A smaller amount would be processed into perbunan at the Bayer factory at Leverkusen. Before dismantling, the Huels works produced 3,100 tons of butadiene a month and could produce about 4,600 tons of buna a month. Of the original 18 ovens, only four now remain, with a capacity of 1,800 tons a month. About 2,250,000 D-marks and about five months' work will be needed to get the plant going again, it is thought.

In the second stage of development it

is planned to produce butadiene by a new method not needing coal and electricity. About 1,800 tons of butadiene and 1,800 tons of buna a month could then be produced. Some 15,000,000 D-marks would be needed; the new plant should be able to start production in about 18 months' time. The management of the Huels works believe that they can produce a product whose price would be substantially below the world price of natural rubber.

The capacity of the Bayer works at Leverkusen has been increased as a result of Bayer's taking over some decartelised I.G. Farben plants. The number of workers has risen from 12,000 pre-war to 18,000 now. But the perbunan plant was partly dismantled, and is also out-of-date. The management of Bayer's refuse to say when production of perbunan will be resumed. Negotiations were taking place with Huels on the supply of butadiene, it was stated. 'Financial circles' had also been approached for capital for the perbunan plant. With enough money, coal and butadiene assured, Leverkusen could produce about 100 tons of perbunan a month, rising to 500 tons a month within two years. The entire European demand for perbunan would then be covered.

The coal situation at Leverkusen has been temporarily relieved by the supply of between 20,000 and 30,000 tons of United States coal. At present Leverkusen produces mainly aniline dyes, textile auxiliaries and chemicals. About 30% of production is said to be exported.

The greatest buna producer in all Germany today is the Schkopau plant in Saxony, Soviet zone. The East German authorities have never disclosed production figures for this plant, the biggest in Germany, but reports reaching Berlin indicate that buna output has now reached 40,000 tons p.a., compared with 60,000 before the war. East Germany's five-year economic plan provides for a further 'considerable' increase in production at Schkopau.

Buna production there was resumed immediately after the war ended. The plant, slightly damaged, was converted into a Soviet-owned company after a small part of it had been dismantled and sent to the Soviet Union as reparations. Most of the buna produced is going to the Soviet Union and the East European states.

**Laboratory driers.** There are many occasions when a supply of dry air is required in laboratory work, for example, humidity control in drying cabinets, prevention of condensation in cooling chambers, drying out vessels and tubing, and removal of oxidising moisture from furnace atmospheres. The portable Birlec *Lectrodryer*, described in a new leaflet, is claimed to meet such requirements economically. It operates by adsorption on activated alumina, which can be reactivated *in situ*. Air and many common gases can be dried as well as certain organic liquids.

# Radioactive Isotopes in Metallurgy

As in all industries, the majority of investigations reported in which radioactive isotopes have been used in metallurgy are on the laboratory scale, and involve following the movements of particular atoms. Only a few applications have been on a factory or plant scale, not necessarily because of any lack of interest in or use for radiotracing methods, but to some extent because of the increased expense and health hazards involved in employing these methods on a large scale. It is hoped that the metallurgical industries will be encouraged to make greater use of the unique advantages that these techniques give, and the reported investigations have been classified with that end in view.

## Diffusion and self-diffusion

**R**ADIOACTIVE iron ( $Fe^{55}$  and  $Fe^{59}$ ) has been used in diffusion studies with particular reference to steel. The investigations were designed to establish the diffusion rates that enter into such phenomena as annealing, hardening, recrystallisation, grain growth, creep, and high temperature behaviour. Since diffusion in the solid state is a vital process in the general behaviour of metals, radioactive isotopes offer a particularly suitable means of investigating the quantitative aspects of the problem.

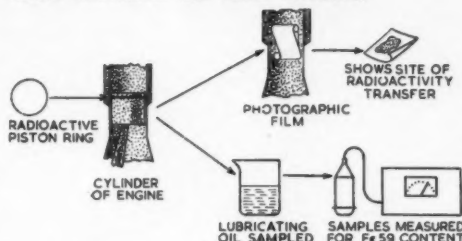
Determination of the diffusion of carbon in alpha (body-centred) iron has been attempted with radioactive carbon ( $C^{14}$ ). Of theoretical importance because of its bearing on the ageing of steels, the magnetic after-effect and the elastic after-effect, such a determination is of practical importance in the decarburisation of plain carbon and alloy steel.  $C^{14}$  was introduced into iron by a pack carburising method employing a mixture of charcoal and labelled  $BaCO_3$ . Although experiments by autoradiography to determine microstructure were unsuccessful, experimentation was continued to determine the diffusion coefficient of  $C^{14}$  in alpha iron at temperatures below  $725^\circ C$ .

Radioactive zinc and copper have been used to study self-diffusion in stressed metals, the techniques being similar in each case. Single crystals of the metal are electroplated with the isotope, and then placed under stress in a furnace at temperatures approaching the melting point of the metal. When the isotope has had sufficient time to diffuse into the crystal, the specimen is removed and sectioned. The activity of the sections is measured to obtain the amount of diffused material at various distances from the original plated surface. From this, the diffusion coefficient and the activation constant are obtained, and the effect of the stress on these quantities ascertained. It has also been suggested that the question as to whether the difficulty of soldering copper that has been tinned with a thin layer, and left for some time, is due to diffusion of tin into the copper might be answered with the aid of radio-tin.

## Properties of alloys

Radioactive tungsten ( $W^{185}$ ) has been used to determine the location of tungsten in a 70% nickel, 25% chromium, 5% tung-

Fig. 1. (right). Schematic diagram of components in the autoradiograph process as used in corrosion studies: A, metal base. B, radioactive plating. C, photographic plate. D, oxide formed where corrosion has been initiated.



sten alloy. By the use of autoradiographs in conjunction with photomicrographs, it was shown that the tungsten is located in the dendrites. In a cobalt-base alloy, it was shown that the tungsten is generally associated with the inter-metallic compounds and solid solutions between the dendrites.

Silicon rectifiers have a few tenths per cent. of aluminium mixed with the silicon, and it is of interest to know whether the Al is distributed uniformly through the silicon. If a silicon-aluminium specimen is irradiated with neutrons, the silicon atoms either do not become radioactive, or else the activity quickly disappears on removal of the specimen from the neutron source. Some of the aluminium atoms, however, are converted to sodium atoms with a half life of 14.8 hr. By autoradiographic means, the distribution of the aluminium atoms may conveniently be discovered. Autoradiographic means have also been employed with radioactive phosphorus ( $P^{32}$ ) to measure the amount and location of phosphorus in alloys.

## Corrosion effects

Radioactive iron ( $Fe^{55}$  and  $Fe^{59}$ ) has been employed to investigate the corrosion of iron. The radioactive material is used in an electroplating solution made up in the usual manner, and a polished test surface is then plated with a thin layer of the radio-iron. After a good original autoradiograph has been obtained—to enable any initial electroplating defects to be noted—the sample is exposed to corrosive attack for a set length of time (Fig. 1). The surface is then blown dry by compressed air, care being taken to see it is not marred, and then placed in contact with a photographic plate for three days, applied pressure being desirable to increase the clarity of the image.

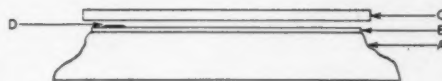


Fig. 2. (left). The method by which radioactive piston rings ( $Fe^{59}$ ) are used to determine engine wear. Particular advantages are that the transfer of metal may be measured to 1/100,000 oz., that the oil is sampled during operation of the motor, and that the location of the wear is shown by developed photographic film.

Development of the plate enables the corrosion process to be seen in considerable detail. The spots of oxide formed at the beginning of the corrosion process cause the dilution of the iron with oxygen, thus reducing the degree of concentration of radioactivity, and consequently the density of the developed plate at this point. In addition, the layer of oxide absorbs part of the radiation from the underlying iron. By using a very thin plating of the active material, slight points of corrosion break completely through the radioactive layer, and produce a high change in activity at the surface by even slight pitting of the iron.

The study of minute differences on the surface of metals and their influence on the way in which corrosion occurs has been carried out with various isotopes. In one case, stearic acid was tagged with  $C^{14}$ , so that after application to metal the disposition of the acid molecules on the metal surface could be traced.

## Metal cleaning

A radioactive tracer technique applied to metal cleaning studies proved to be considerably more sensitive than existent methods for estimation of soil removal, and to be quantitative in character.  $C^{14}$  tagged N, N-di-n-butyl stearamide was synthesised, and added to the lubricating oil used in the evaluation of metal cleaners, in the proportion of 1 : 97. After the soil had been applied to the metal surfaces, they were cleaned by immersion in several baths of a 5% Santomerse No. 1 95% anhydrous sodium metasilicate solution in a concentration of 5% by weight in distilled water. The metal plates were assayed for the radioactive contamination remaining after each bath, and the limit of sensitivity attained was said to be  $2 \times 10^{-9}$  g/cm<sup>2</sup>, as

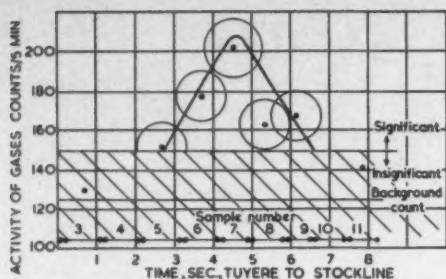
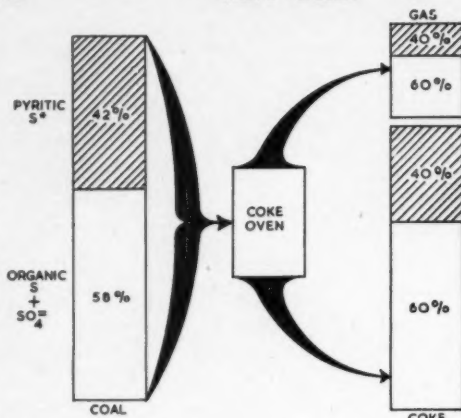


Fig. 3. (left). After radioactive gases have been introduced into a tuyere, the gas samples from the furnace are analysed, and the times of transit may then be shown graphically.

Fig. 4. (below). Schematic diagram of behaviour of sulphur in a coking operation. Pyritic sulphur ( $S^*$ ) has been labelled with  $S^{32}$  tracer.



compared to a sensitivity of  $1 \times 10^{-4}g$ . usual with gravimetric techniques.

### Friction studies

Several friction studies have been reported based either on the assaying of lubricants for radioactive metal traces, or on using autoradiographic means for determining the exchange of material at sliding surfaces. In an example of the first type of investigation, machinery bearings were irradiated in a neutron pile, and it was possible to estimate the bearing wear to  $5 \times 10^{-5}$  in. In other studies, irradiated piston rings (Fig. 2) and radio-chromium plated surfaces were used. In all these cases a very much smaller amount of worn metal could be detected than with conventional techniques.

In another investigation, the chemical reactions occurring at a lubricated surface were studied with radioactive iron. Any iron which reacted with the oil, or chemical additives in the oil, could be measured with a sensitivity of 1 pt. in 10 m., in as little as 20 mgm. of oil.

### Electroplating

Two interesting applications have recently been suggested. The rate and method of growth of a surface layer being laid down electrolytically might be studied by autoradiography, particularly in the early stages. Tracers might also be used to follow the depletion of minor constituents in electroplating baths.

### Factory-scale investigations

From both chemical and physical standpoints it is desirable to know the gas velocities obtaining in various positions in a blast furnace in order to determine with certainty the effects of burden porosities and the times of contact between the gases and solids. The method followed was for a small volume of radioactive gas to be introduced into a tuyere, and for gas samples taken from a point in the furnace to be assayed, the times of transit being calculated from the results obtained. It was necessary to use an inert gas such as radon or xenon, so that it could pass unchanged through the high

temperature zone: radon was in fact used, as it was more readily available. 8 mc. of radon were blown into the tuyere by explosive means, and gas samples from the centre of the stockline taken into a number of gas jars over a 22 sec. period. After assay of the various containers, it was shown that the peak activity at the centre of the stockline occurred about  $4\frac{3}{4}$  sec. after the radon was introduced, and that activity extended over the range from  $2\frac{3}{4}$  to  $6\frac{3}{4}$  sec. (Fig. 3).

The largest experiment on an industrial process so far reported was carried out by a steel company. One of the most im-

portant sources of undesired sulphur in steel is the coke used in the blast furnaces. Because sulphur in coal is mainly in two forms, pyritic and organic, and the ratio of these two forms varies on different coals, it was desirable to know which type of sulphur in the coal was carried over into coke. If one form was preferentially eliminated in the coking process, it would be possible to exercise some control over the product by judicious choice of starting material. Because the conditions existing in a coke oven cannot be readily duplicated in the laboratory, a full scale plant test was carried out.

Pyrites containing radioactive sulphur ( $S^{32}$ ) were synthesised in the laboratory, and then intimately mixed with 12 tons of coal, which represented a single charge in the coke oven. During the coking operation, gas evolution was carefully measured, and the total and radioactive sulphur content of the gas was determined at regular intervals. After the coke had been discharged, representative samples of the product were also analysed for total and radioactive sulphur. The results showed that the ratio of radioactive (pyritic) sulphur to total sulphur was the same in both gas and coke, and was equal to the ratio of pyritic to total sulphur in the original coal. This was interpreted as meaning that all the sulphur in coal is equivalent in the coking process, and that the ratio of pyritic to organic sulphur in the coal does not influence the sulphur content of the finished coke (Fig. 4).

The distribution of phosphorus between slag and steel has been studied by tracer methods. Calcium phosphate containing radioactive phosphorus ( $P^{32}$ ) was synthesised and added to the slag. The rate of appearance of  $P^{32}$  in the steel was a measure of the rate at which phosphorus reached equilibrium between the two phases. Combined with a chemical analysis for total

(Concluded on page 385)

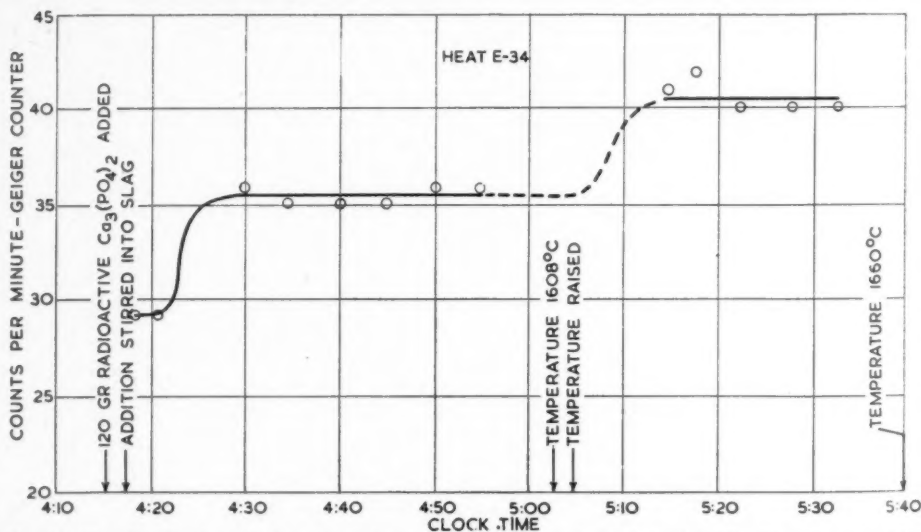


Fig. 5. The distribution of phosphorus between slag and steel studied with  $P^{32}$ . The rate at which P reached an equilibrium distribution between two phases is readily determined, and the effect of temperature on equilibrium is also shown.

# The British Experimental Pile

## FIRST FULL DETAILS OF CONSTRUCTION AND OPERATION

Here is the first detailed description of the construction and composition of Bepo (British-Experimental Pile), the larger of the two piles at the Atomic Energy Research Establishment at Harwell. Release of this information by the Ministry of Supply follows the recent agreement between the atomic energy authorities of Canada, the United Kingdom and the United States on publication of certain information. The fundamental design of Bepo was undertaken by the U.K. staff working at the Montreal Laboratory in Canada during 1945-46. The Department of Atomic Energy at Risley, who were responsible for the engineering design and construction, began work on the project early in 1946. The Ministry of Works, under the direction of the Department of Atomic Energy, began construction in June 1946, and the work was sufficiently far advanced in two years for the plant to be handed over to the Harwell Research Establishment for final tests and operation.

**B**EPO is a graphite-moderated, air-cooled pile with a central thermal neutron flux of  $10^{12}$  neutron/cm.<sup>2</sup>/sec., a critical loading of 28 tons of uranium metal, and a full uranium load of 40 tons giving rise to an excess reactivity which can be used for producing isotopes or for experimental purposes. The air-cooling system was designed to limit the maximum surface temperature of the fuel elements to 200°C. Loading to critical size took place on July 5, 1948, the uranium being added channel by channel until the reaction became divergent. The power of the pile was continuously monitored in the sub-critical state by specially made measuring instruments and, when the pile diverged, the power was permitted to rise to 50 W, with no coolant flowing, before the reaction was shut down by the automatic safety system. This was followed in the ensuing months by a gradual work up to full power, the designed maximum flux being reached by the Christmas of 1948.

As Harwell was established in a vacated aerodrome which included four large hangars, it was possible for the construction of the pile to proceed at once under cover by using one of them as the pile building, although a section of the roof had to be raised 20 ft. to provide additional head room above the pile for handling tall experimental equipment. The cooling air exhausters required a new building and this was constructed adjacent to the selected hangar to which it is connected by underground air and service ducts.

A considerable volume of air is required to cool the pile (about 180,000 cu.ft. min.) and this is drawn through the pile, in a single-pass system, by a number of exhausters situated between the pile and the main exhaust stack. Filters are installed in the air intake to clean the air before it is passed through the pile and another set between the pile and the compressors to remove any trace of radioactive dust which might arise in the pile itself before the air reaches the main machinery which discharges into the base of a 200-ft.-high stack.

Since materials introduced into the pile are radioactive on removal, there is within

the pile building a concrete storage block with accommodation for some 5 tons of irradiated uranium cartridges (usually called 'slugs') and numerous other cells where irradiated materials can be stored temporarily on removal from the pile until they have decayed sufficiently for safe handling. Irradiated material from the pile has to be withdrawn direct from the pile into lead flasks and a feature of the pile building is the overhead crane system to permit of the easy handling of these heavy flasks.

### General construction

The general construction of the pile itself is shown in the diagram. The reacting part is a cube of graphite of 26-ft. side through which pass 1,849 channels about  $3\frac{1}{2}$  sq. in. in cross-section and pitched at  $7\frac{1}{4}$  in. centres. The central 900 of these channels can accommodate bars of uranium machined into cylindrical rods, 12 in. long by 0.9 in. diam., encased in aluminium to prevent oxidation of the uranium and the escape of fission products into the cooling air stream.

Twenty uranium bars are included in each channel, leaving 3 ft. of empty channel at each end. The channels are loaded radially outwards from the centre to form a right cylinder of 10 ft. radius and 20 ft. long. The remainder of the graphite around this central core serves as a reflector to neutrons which would otherwise escape from the central system.

### Graphite moderator

In addition to the main channels, this graphite moderator is perforated with holes for experimental purposes, so that the actual structure is somewhat complicated. It is built up of standard blocks each  $7\frac{1}{4}$  in. square by 29 in. long, with features machined in their surfaces which provide when assembled all the necessary holes. In a workshop specially erected for the purpose at Harwell, 850 tons of graphite were machined into some 25,000 blocks with more than 60 variations in the basic types. The general machining tolerance adopted was  $\pm 0.0025$  in. on all dimensions and such accuracy was maintained that the

total height of the 26-ft. completed stack was correct to within 0.03 in. From the top and one side cubes of graphite 'thermal columns' project from the main moderator. These columns, by virtue of their distance in graphite from the nearest source of fast neutrons, provide a source of almost pure thermal neutrons.

The 900 central channels are connected at both the air inlet and air outlet ends by headers 4 ft. wide and extending over the whole graphite face. The headers are connected to the 10 x 8 ft. underground ducts of the main air system. The main outer structure of the pile consists of a reinforced concrete and steel shell lined with 6-in.-thick cast-iron plates, and constitutes the biological shield for attenuating the intense radioactivity within the pile. The radiation level outside when the pile is running at full power is only twice that which exists when the pile is fully shut down, thus ensuring that the sensitive instruments used for experimental work are not adversely affected by background activity.

The 6-in. cast-iron shield provides an initial large attenuation of thermal neutrons and gamma rays, so that the bulk of the radiation energy escaping from the reflector is converted into heat in a conducting and heat-resistant material, from which it can be easily transferred to the cooling air stream circulating past the sides of the cast iron, so avoiding high temperatures in the concrete.

### Assembly of pile shield

When the graphite moderator was stacked, stringent precautions were taken to ensure clean conditions, so as to exclude dust and other impurities with high-capture cross-section for neutrons which might prevent the pile reacting. To obtain these conditions (not usually available on a construction site), the shield was built as a complete box, the air ducts and all holes through the shield sealed off and the inside of the pile meticulously cleaned and lightly pressurised with clean air before graphite stacking was started. The entire shield, therefore, had to be constructed as a unit and one of the problems

in the construction of the pile was the extreme accuracy with which the pile shield comprising some 3,000 tons of concrete and 600 tons of steel had to be assembled in order that continuity and alignment of the 3,800 holes common to both graphite and shield was preserved.

The method adopted was first to level and set the cast-iron base plates on which the graphite is stacked to within  $\pm 0.01$  in., then to erect on this base the steel frame structure of walls and roof. On this structure the 6-in. cast-iron shield plates and the tubes through the shield were set to  $\pm 0.01\%$  in 3-ft. 'lifts,' each lift being concreted in turn, great care being exercised to ensure that the concrete penetrated to all interstices in the shield and that no voids or cracks were left to provide subsequent leakage paths for radiation.

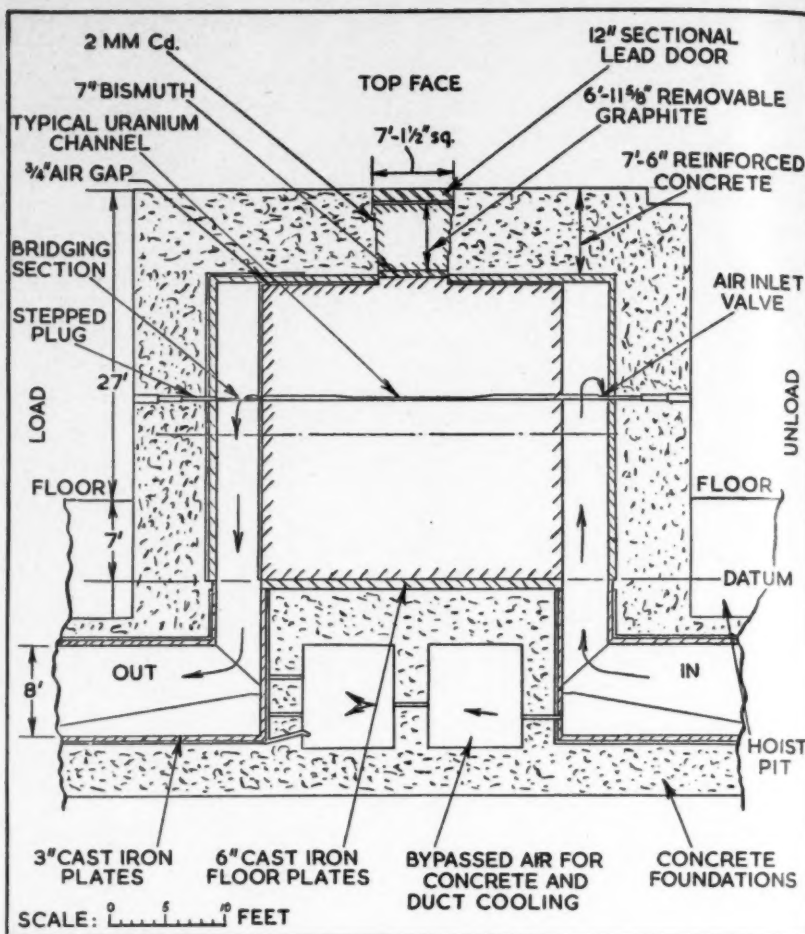
### Loading

The pile is loaded by pushing in a row of slugs from the charge face of the pile, and unloaded by means of steel push-rods which push the irradiated uranium slugs out through the unload face into a lead-shielded container. A bridging section is provided connecting each uranium air channel with a hole through the shields so that the uranium bar can cross the air headers. The holes through the shield are, of course, plugged to prevent the escape of radiation at all times other than when an individual channel is being loaded or unloaded. It can be seen from the diagram that all holes through the shield are stepped so that no direct path is provided for escape of radiation. Loading and unloading machines are carried on 12-ton hydraulic hoists extending the width of the pile and can be positioned accurately to any horizontal row of shield holes on the load and unload faces of the pile and traversed on the hoists to select the requisite hole in a given horizontal row.

### Cooling system

As explained earlier, the pile is cooled under induced air draught, which was preferred to forced draught in order to establish a negative pressure within the pile with respect to atmosphere to ensure that any leakage of air at the many holes through the shield wall would involve fresh air inwards and not activated air or dust outwards. Although this results in less advantageous conditions at the exhausters than a forced-draught system, it is fully justified in practice by the greater simplicity of the experimental arrangements.

The basis for the specification of the cooling system was the air quantity and pressure drop required through a central uranium channel in order to keep the temperature of the hottest uranium cartridge below  $200^{\circ}\text{C}$ . for a rate of heat release corresponding to a total power of 4 MW. The heat release per unit length along a channel and from channel to channel across the pile is not uniform, since the distribution of neutrons in a pile



Longitudinal section of the British Experimental Pile.

is a maximum in the centre falling off towards boundaries. In order to economise in pumping power the air flow down each channel is adjusted to suit its heat release.

Five 1,400-h.p. single-stage compressors running as exhausters are installed to draw the air through the pile, of which four are required for the full power conditions. The drive for each exhauster is from a direct-coupled 11,000-volt three-phase induction motor supplied from the national grid through suitable step-down transformer and control gear.

### Control system

The pile is controlled by introducing four boron carbide-filled hollow steel rods, 2 in. in diam., horizontally into the side of the pile so that, when fully inserted, the rods extend across the reacting core. These rods can be accurately positioned from push buttons in the control room. In addition there is a second system of ten similar rods, known as shut-down rods, operating vertically in the pile and having two positions only, right in or out.

When both systems are fully inserted the fission rate within the pile is about a milliwatt, since the boron in the rods

absorbs thermal neutrons very strongly. The start-up procedure is first to withdraw the shut-off rods, then the control rods to a position where it is known that the pile power will rise at a reasonable rate; when the desired power is reached the rods are moved in until the indicated power level is steady. In subsequent operation, compensation for self-induced changes in the reactivity of the pile must be made by control rod movement. These changes occur due to variation in the graphite and uranium temperatures and the build-up of certain fission products with a high neutron capture cross-section. All the self-induced reactivity changes tend to shut down the pile, so that subsequent to start-up a continued outward withdrawal of the control rods is necessary for several days until steady conditions are reached. This adjustment was originally effected by hand, but an automatic regulating system has now been installed which not only relieves the operator of a tedious task but also maintains a more steady power level than can be achieved by hand control, a desirable feature for experimental work.

The shut-down rods, controlled by com-

pressed air, are available at all times to shut down the pile rapidly in under a second if conditions of fault should arise in the plant.

The power of the pile is measured in two ways, first by means of a heat balance measurement in the cooling air. This, although accurate, has a slow response, due to the inherent thermal time lags of the system, and therefore is no use for the instantaneous control of the pile. The second is by measuring the thermal neutron flux. This method, which is sensibly instantaneous, employs ionisation chambers within the pile shield which produce a small electric current proportional to the thermal neutron flux in which they are immersed. This output is amplified and displayed on suitable instruments in the control room.

In the control room are instruments indicating activity gained by cooling air on passing through the pile, while gamma and thermal neutron health monitors, permanently installed around the pile, are connected up in a similar way.

In addition to the many lessons learnt during the design, construction and initial

start-up of the pile, in two years of full power operation Bepo has been a valuable research tool. All the facilities provided have been exploited, neutron beams from the thermal columns have been used in many nuclear experiments, while physicists, chemists, metallurgists and biologists have been able to further their knowledge of the effects of radiation on matter. The rapidly growing demand for radioactive isotopes in all fields of scientific research and for industry has been met by production in Bepo, the largest radioactive source so far handled being a 500-curie cobalt source for biological work. In the technological field experience is being gained which has direct application in the development of nuclear power, important aspects being the development of control equipment, observation of the effects of radiation on structural materials and the study of shielding design.

In all, Bepo is playing a vital part for Britain in the building up of fundamental knowledge of the science and engineering of nuclear energy and in providing new tools for research in many other fields of scientific endeavour.

phenolic resins have been published but, according to Seymour, none is adequate. The most satisfactory results to date have been obtained by mixing liquid phenolic resins with fillers containing setting agents in accordance with manufacturer's directions, casting test wafers measuring  $1 \times \frac{7}{8} \times \frac{1}{4}$  in. and curing at 90° F. for 24 hr. followed by 3 days at 140° F. Comparative corrosion tests were made using the wafers as test pieces. Considerable information was also obtained by noting etching, loss in strength, embrittlement, shrinkage, curling, etc.

Results showed that temperature has no deleterious effect on the resistance of either silica or carbon-filled phenolic cement to 25% sulphuric acid, 50% sulphuric acid, saturated aqueous sodium chloride, mineral or vegetable oil. Both types are resistant to concentrated sulphuric acid and nitric acid at room temperature, but are attacked at higher temperatures. Both are fairly resistant to 5% sodium hypochlorite at room temperature but are slightly etched. There is little change in weight in alkalis but physical properties of the surface are adversely affected at a pH above 8.5.

Phenolic resin cements are not resistant to sodium hydroxide but they are more resistant to concentrated than to dilute caustic. This phenomena explains why phenolic resins have been used successfully as jointing materials for floors in the presence of highly concentrated alkali at ordinary temperatures.

The chemical resistance of phenolic resins is a function of the ratio of formaldehyde to phenol used in the preparation of the liquid resin, pH of the initial and final condensation, temperature of resin formation, chain length of the molecule and chemical constitution of fillers or diluents.

Phenolic resins, like alloys, often prove to be serviceable in the presence of slow attack by chemicals. However, unlike alloys no method is available for the estimation of useful life. Phenolic resin structures have been used in the presence of sulphuric acid in Germany for over 20 years and in the U.S.A. for over 10 years.

## Phenolic Resins in Chemical Plant

**A**LTHOUGH commercially available for almost half a century, phenolic resins were not used as materials of chemical construction in the U.S.A. until fairly recently. On the other hand, chemical equipment consisting of mixtures of acid-washed asbestos and phenolic resins was manufactured in Europe over 20 years ago under the trade name of *Faolite*.

As a result of their outstanding resistance to solvents, salts, and non-oxidising mineral acids, coatings, chemical equipment and cements derived from phenolic resins are now being used widely in the chemical, steel, paper, petroleum, food, textile and plating industries. Applications of phenolic resins in this field as well as test procedures for determining the chemical resistance under different conditions were described by R. B. Seymour in a paper presented before the Paint, Varnish and Plastics Chemistry Division of the American Chemical Society and published in *Corrosion*, 1951, 7 (5), pp. 151-155.

While filled resins have been proposed for use as coatings, most phenolic coatings consist simply of an alkaline catalysed phenol-formaldehyde resin which is applied as an alcohol or ketone solution and cured by the application of heat. This is the largest use of phenolic resins in the field of chemical resistance but is being replaced to some extent by thermoplastic coatings based on vinyl chloride copolymers. Phenolic coatings have excellent resistance to solvents, salts and non-oxidising inorganic acids but are inherently brittle and therefore should not be subjected to heat or mechanical shock. Coatings made from

alkylphenols and condensation products of phenol with vegetable oils are more flexible but less resistant to chemical agents than straight phenol-formaldehyde resins.

Pipe and equipment produced by polymerising an alkaline catalysed liquid phenol-formaldehyde resin filled with acid-washed asbestos or carbon have been used in the U.S.A. for over 15 years and for almost twice as long in Europe. Provided sufficient wood or steel reinforcement is supplied, the size of such equipment is almost unlimited, but the largest unsupported structure known weighs about 2 tons. One estimate of the maximum dimensions gives the limits for the diameter as 9 ft., the height as 12 ft. for cylindrical tanks and 12 ft. square for rectangular equipment although rectangular units may be joined together to make equipment of almost infinite length.

Phenolic resin reinforced with *Fibreglas* textiles is now being used to protect ceramic equipment and for the production of pipe and chemical apparatus.

At present there are a large number of distributors of chemical resistant cements based on furfuryl alcohol polymers and phenol-formaldehyde resins, but very few firms produce their own liquid resins. Phenolic resin cements consist of a mixture of an alkaline catalysed phenol-formaldehyde liquid resin having solids contents of at least 70% and a minimum convertible solids content of 90% with a corrosion-resistant filler. Silica, asbestos, barium sulphate and carbon are the most widely used fillers.

Tests for the chemical resistance of

## Radioactive Isotopes in Metallurgy

(Concluded from page 382)

phosphorus, the value of the equilibrium constant could be obtained, while the factors controlling rate and equilibrium could be investigated by appropriate variations of temperature, calcium-phosphorus ratio, etc. (Fig. 5). It has also been suggested that radiotracers could be used to measure the amounts of valuable materials passed out in slags from smelting processes.

Another suggestion is that the flow of metal during extrusion, casting, or forging processes might be studied if layers of radio element were introduced into the original metal block (by, for example, continuous casting methods) and then sections of the metal autoradiographed.

# Work of the Government's Fuel Chemists

## Pyrites

TO augment the output of home-produced, sulphur-bearing materials for sulphuric-acid manufacture during the war, the Coal Survey made an extensive survey of colliery refuse to find materials sufficiently rich in pyrites to justify concentration of the mineral. Prior to the war, the main home-produced materials used by the sulphuric-acid industry were anhydrite and spent oxide, but about 3,000 tons annually of hand-dressed, lump pyrites produced by the collieries were used in hand-fired kilns. During the war this production was increased to about 10,000 tons p.a. The most important development, however, was the erection by the Ministry of Supply of eight concentration plants to produce pyrites fines from colliery refuse, which resulted in the production of fines at the rate of about 25,000 tons p.a. The first of the new plants did not start operation until July 1941, but from then until the end of 1945 the total production of lump and fine pyrites amounted to about 120,000 tons. In the crushing of the refuse prior to treatment in the concentration plants a certain amount of coal is liberated; although of small size the material was of considerable value, in view of the general coal shortage, and arrangements were made to segregate it. As a result, the amount of coal recovered from the eight pyrites concentration plants reached 50,000 tons p.a.

## Chemical examination of coal

The investigation of the constitution of coal was discontinued during the war, but later the work was resumed. The addition of ethylene to coal in the presence of catalysts at 330°C. and under pressure has been investigated. The ethylene which is consumed appears in the product partly as an oil (polymer) which is readily extractable from the solid product by light petroleum, and partly as a solid, ethylene-coal, addition product. Carbonisation of this latter material showed higher yields of coke and gas than did the original coal.

A new type of furnace has been constructed in which a controlled amount of preheated air is introduced at the furnace top, reducing contamination to a minimum, an important factor in the accurate determination of ash in coal.

A method for the determination of the small amounts of fluorine in coal was developed. The fluorine content of a wide range of British coals was found to vary from nil to 140 p.p.m. It has been established that when the coal is burnt almost all the fluorine is volatilised and evolved probably as hydrogen fluoride or as silicon tetrafluoride. This fact has an industrial significance, because both of these substances have a deleterious effect upon the brickwork or metal parts of furnaces. If a high-fluorine coal is used in

A surprisingly wide range of researches was carried out during the late war by chemists of the Government Fuel Research Station. Details of this work, not released at the time for security reasons, have now been published.\* Here is a selective summary of these investigations, ranging from the production of pyrites to the gasification of coal, and from the manufacture of hydrogen to the extraction of wax from lignite.

a direct-contact type of kiln, as used, for example, in melting or dehydration of foodstuffs, the products may contain injurious amounts of fluorine.

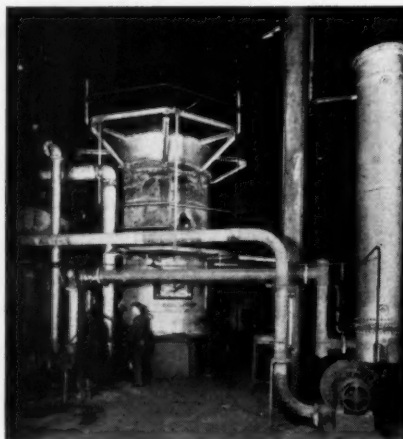
## Gasification of coal

Experiments were carried out on the gasification of bituminous coals by means of steam and oxygen in a reaction chamber in which a vortex was produced by the flowing stream of gases, with the object of designing a plant which would operate under pressure to produce a gas similar to that obtained in the Lurgi process. The best result gave a yield of gas equivalent to 94 therms/ton of coal gasified. The rate of gasification of the coal was 0.66 lb./hr., with a chamber temperature of 898°C., oxygen rate of 8.3 cu.ft./hr. and steam rate of 4.5 lb./hr. The time of contact between air and steam was 0.4 sec.

## Production of hydrogen

Hydrogen was produced at the Fuel Research Station before 1939 for research work on the hydrogenation of coal and

\*Fuel Research, 1939-1946. H.M.S.O., 1950. Pp. 69. 3s.



Hydrogen-generating plant.

tar, and on the production of hydrocarbon oils by synthesis. The plant, first erected in 1926, consisted of four mild-steel vertical retorts, externally heated to about 780°C., and had a capacity of approximately 40,000 cu.ft./day of hydrogen. The capacity was subsequently increased to 70,000 cu.ft./day by increasing the number of retorts to six and by improvements in the method of heating and in heat recovery. In 1940 a second plant was erected and operated for the Air Ministry. This plant contained 12 mild-steel retorts of a somewhat larger size than the first plant and had a rated output of 150,000 cu.ft./day of hydrogen.

Early in 1941 the Ministry decided that mild-steel retorts were not suitable for the continuous production of hydrogen under gas-works conditions and the Director of Hydrogen Plants arranged for the installation in different parts of the country of a number of hydrogen plants based on the B.A.M.A.G. and Messerschmidt types, suitable for working at operating temperatures up to about 1,000°C.

## Synthesis of methane . . .

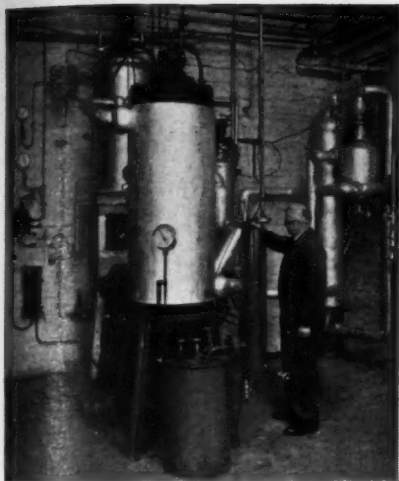
The investigation of the synthesis of methane from gases containing carbon monoxide and hydrogen was continued during the war. The reaction was originally studied as a means of increasing the calorific values of gases made in complete-gasification processes, but during the war attention was concentrated on the catalytic production of methane from water-gas.

## . . . and other hydrocarbons

The study of the Fischer-Tropsch process at the Fuel Research Station was begun in 1935, primarily for the production of lubricating oils by treatment of the primary reaction products. A semi-technical-scale plant of 40-l. catalyst capacity was erected and operated, using the cobalt-thoria-kieselguhr catalyst described in the publications of Franz Fischer and his co-workers. During the war years the work was restricted to a laboratory scale, and a more comprehensive examination of the variables which affect catalyst activity and life and the composition of reaction products was carried out. Work on the synthesis of lubricating oils was continued and a more complete study of the production of fatty acids by oxidation of the primary products was undertaken. The information thus gained advanced the knowledge of this process far beyond what was known from the published literature and was of particular value in the planning and carrying out of the investigations of the Fischer-Tropsch industry in Germany in the spring and summer of 1945.

## Toluene manufacture

Toluene requirements were met during the war by increased production from



**Hydrocarbon-synthesis plant**

*This and the photograph opposite are from 'Fuel Research 1939 to 1946' and appear by permission of the Controller of H.M. Stationery Office.*

coke-oven and gas-works benzole and by imports from the U.S., where production from petroleum was carried out on a large scale. However, it was uncertain whether sufficient toluene would be available from these sources and, at the request of the War Office and, later, the Ministry of Supply, research on methods of producing toluene from other indigenous materials was undertaken. Whereas it was shown that toluene could be produced from benzene, xylene, naphthalene, cresol and *n*-heptane, the efficiency of the conversion varied considerably. The process of most practical importance proved to be the conversion of cresol to toluene by catalytic hydrogenation and a continuous process was developed to a semi-technical scale of 120 gal./day.

#### **Sludging and corrosion in benzole-absorption plants**

An investigation of sludge formation in benzole-absorption was directed mainly to an examination of the effects of the addition to fresh wash oil, chiefly petroleum gas oil, of substances similar to those contained in the coal gas-scrubbed. It was found that the sludge formation is probably due mainly to the oxidation of unsaturated hydrocarbons such as indene to form peroxides, which then undergo numerous secondary reactions. The rate of oxidation is sensitive to the influence of numerous catalysts which may have a promoting or inhibiting effect.

#### **Incendiary products**

Experimental investigations were carried out on incendiary materials for many purposes, but the work was distinguished from studies of pyrotechnics in that a petroleum oil was almost always a major constituent of the type of mixture examined. New types of incendiary mixture were devised. Other work included the investigation of the inflammability of pulverised coal pro-

jected in the form of a cloud, and the development for special purposes of incendiaries based on a mixture of oil and magnesium. The most important work carried out was the study of the preparation and properties of fuel for flame-throwers, fougasses, and flame and smoke barrages.

#### **Emulsion process for solidification of liquid fuels**

Early in 1942, a new process was devised which can be operated in two stages to give two distinct types of products. The first type comprises 'solidified' organic liquids, the use of which greatly facilitates storage and handling and reduces fire hazard. The second type comprises low-density expanded materials, which are suitable for thermal insulation, light-weight 'sandwich' construction and filters.

#### **Wax from lignite**

At the request of the Non-Ferrous

Minerals Development Control of the Ministry of Supply, the possibility of obtaining an indigenous wax to replace imported montan wax was studied. The wax obtained by the extraction of Devon lignite with benzene (yield 5%) resembled montan wax and could replace the latter in certain cases. English and Scottish peats yielded 3 to 15% of wax. The peat waxes had lower melting points and higher acid and ester values than lignite wax or montan wax.

#### **Acetylene substitutes**

A series of tests showed that acetylene for cutting purposes can be satisfactorily replaced by propane or coal gas, with or without enrichment with ether. Acetylene gives faster heating and cutting than any of the other gases and needs less oxygen. The differences found between propane and propane-ether were too small to justify the much higher cost of the latter.

## **Ultrasonic Chemical Processing**

**H**IGH-FREQUENCY sound can now be utilised in full-scale processing equipment to speed up such reactions as distillation, extraction, polymerisation, oxidation, coagulation and the like. Hitherto the uses of this so-called ultrasonic energy have been limited in capacity to the test tube and laboratory stage. The necessary equipment was exhibited publicly for the first time at the end of last year in New York and some of the possibilities and problems are discussed in *Petroleum Processing*, 1950, 5 (1), pp. 39-40. This equipment demonstrated the feasibility of extending the size of ultrasonic operations to production scale, which has been made possible through the development of specially formulated barium titanate ceramics.

#### **Accelerating reactions**

The chief function of the ultrasonic energy is to subject chemical reaction to what might be termed 'colossal' shaking. The vibrations set up by an ultrasonic

generator, or transducer, reach a frequency of 1,000,000/sec. As a result, reactions which normally require several hours to complete might be accomplished in a matter of seconds or minutes.

The prime source of ultrasonic force is found in so-called piezoelectric devices. Quartz and similar crystals have such properties. This means their crystalline structure produces controlled vibrations on the application of electric current. Quartz is scarce material and particularly expensive in the large shapes required for industrial-scale use of ultrasonics. A new ceramic has been developed which has a low impedance in comparison to quartz. This means that a very low voltage can produce the same acoustical energy that would require a very high voltage with quartz. Quartz-type generators, for example, necessitate an input voltage of 2,000 V or more, whereas ceramic elements will operate satisfactorily in the range of 50 to 100 V.

#### **Problems to be solved**

As yet, many chemical engineering requirements in the field of ultrasonics have not been met. Chief problems, in addition to the proper size of the generator, include discovering the best operating conditions, designing accessory equipment, determining methods and techniques to be employed and circulating the existing knowledge on the subject.

American petroleum technologists and chemical engineers are of the opinion that this new method has great possibilities but that considerable development is necessary before its application on an industrial scale can be considered economic. The higher cost of electricity compared with other forms of energy is one factor which must be considered.

#### **Contributions and Correspondence**

THE EDITOR welcomes practical articles and notes on chemical engineering and industrial chemical subjects with a view to publication. All contributions, which should be fully illustrated whenever possible, are carefully considered. A preliminary letter or synopsis is advisable. Address such material to THE EDITOR, INTERNATIONAL CHEMICAL ENGINEERING, 17 Stratford Place, London, W.1.

Letters for publication on any of the subjects covered by this Journal are also invited.

# New Plant and Equipment

## Glycerin distillation plant

Plant for the distillation of glycerin employing a patented process has recently been improved by George Scott & Son (London) Ltd. Standard crude glycerin is first dried continuously in a pre-evaporator under a vacuum of 28½ in. The dried crude passes into a forced circulation tubular still of special design, where it circulates through tubes and is evaporated by saturated steam of 150 to 200 lb./sq.in. pressure circulating around the tubes. The absolute pressure inside the still is between 1 and 3 mm. The vapour passes upwards through the large bore tubes in the first, or hot condenser, with the temperature conditions set so that the vapour leaving the top of the condenser is only just below the boiling temperature of pure glycerin.

The condensing vapour evaporates water around the tubes. This water vapour is condensed in an auxiliary condenser—coil or tubular—the control being effected by regulating the supply of condensing water through the auxiliary condenser. The temperature of the water around the glycerin condensing tubes is held as required between 120 to 155°C. The condensed distilled water gravitates back to the condenser.

Any off-test glycerin passing out of the hot condenser is condensed in the second vertical tubular cold condenser. Uncon-

densable vapour or air, etc., is compressed from the high vacuum to the vacuum obtainable by a wet displacement type vacuum pump, say 28½ to 29 in. vacuum, by a steam jet air ejector into the final condenser, which also takes the vapour from the pre-evaporator. The off-test and product from the still residues is returned for redistillation.

It is claimed that the good quality distilled glycerin is practically water-white 99.8 to 99.9% glycerol content—and over 75% of the glycerol fed to the plant will, after a mild bleach and after a single distillation, fully satisfy the B.P. specification for chemically pure glycerin.

The glycerin leaves the hot condenser while boiling, passes through a water-cooled coil or tubular cooler, and is withdrawn from the vacuum system and delivered to store tanks by means of a positive pump.

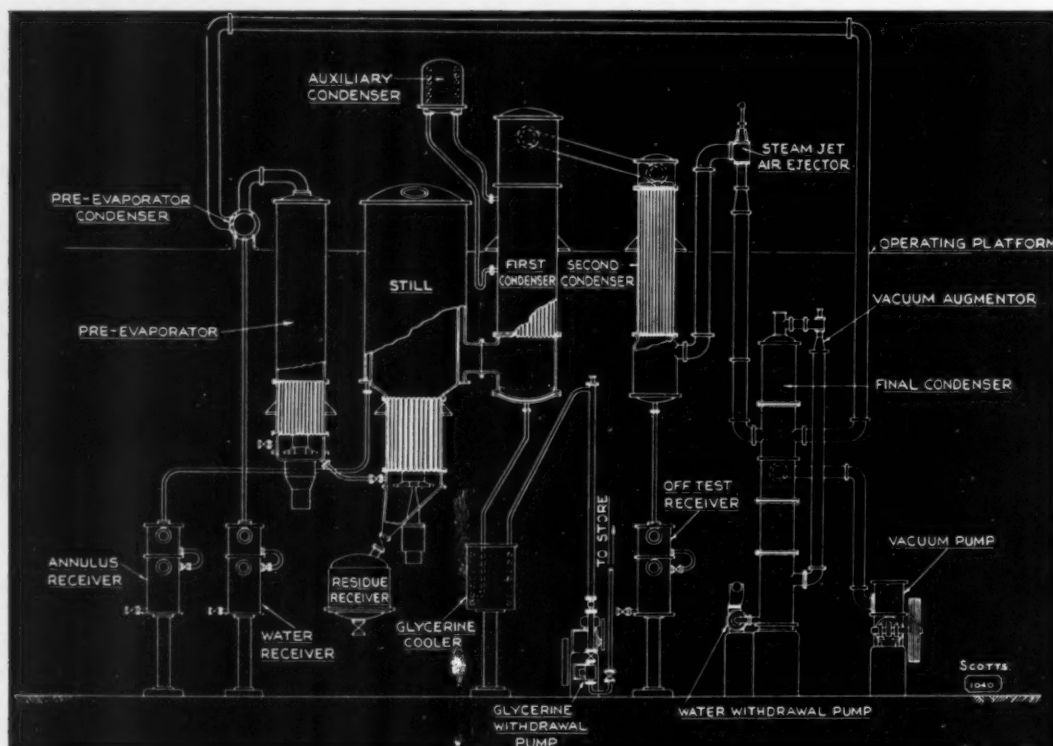
## Glass chromatographic adsorption columns

Many separations are possible by chromatographic adsorption which were previously very difficult to accomplish by classical methods. A range of glass columns is supplied by Quickfit & Quartz Ltd. to cover the main laboratory requirements. Micro columns are available for research work when only small quantities of the material are available. Other sizes are

available for laboratory preparation and purification of various mixtures of solids by chromatography as well as larger models for normal separations. The glassware has been designed to provide equipment which can be assembled quickly and has all the advantages of apparatus assembled from interchangeable units.

The adsorbent material is supported on a sintered glass disc. This provides a flat surface, thus ensuring uniform packing of the column, for which purpose tamping rods are provided. The porosity of the sintered glass disc is such that it will not allow the adsorbent material to pass through, but, if necessary, the column of adsorbent material can easily be extruded by the application of compressed air to the cone at the bottom of the glass column. The columns of two models are provided with an adapter, whereas two others have a sintered disc sealed into the cone to support the column of adsorbent material. This is to enable the column to be extruded by means of a tamping rod if the adsorbed material is not to be recovered by elution with a suitable solvent. To facilitate the percolation of liquid through the columns slight vacuum can be applied. If the nature of the solvent is such that the application of vacuum is impracticable, positive pressure can be used. For this purpose a large cylindrical reservoir of 5-litre capacity, together with a cone adapter, is fitted to the top of the column. Thus, it is possible to run the column under positive pressure for a considerable time, without refilling.

The tap adapter is particularly useful



[Diagram: George Scott & Son Ltd.]

**High vacuum glycerin distillation plant.**

when the apparatus is used with ion exchange resins for separation of solutions of inorganic salts. The granular nature of these resins makes it necessary to restrict the flow of liquid through the column in order to obtain maximum efficiency. The use of standard adapters and dropping funnels greatly facilitates the addition of the solution to the column.

### Sedimentation tank

A New York company has developed a new type of sedimentation tank for hot-process water softeners. Known as the Graver Sphericone, this design is a hemisphere mounted on top of a cone. The inverted cone provides a uniformly increasing area from tip to circular top. The raw water, steam and chemical inlets, spray heater, de-aerator and treated water collected are all mounted in the hemisphere while the inverted cone contains the down-take sludge bed and rising zone of the water being treated.

### Laboratory oven

A new type of hot-air oven with close temperature control, designed by Electricals Ltd., has a temperature range ambient to 300°C and can be used for any temperature within these limits. The thermostat control will give the desired temperature at all parts of the oven to within  $\pm 1\frac{1}{2}\%$  with shelves loaded, although the limits are finer than this for any one point. This very close temperature control is obtained by special features, the principle of these being a hot-air bath, surrounding the interior of the oven, in which the air is circulated by a fan. An additional fan agitates the air in the oven interior. The heat insulation is such that the exterior of the oven remains cool at all times. The small wattage controlled by the thermostat in this oven, plus the extra elements controlled by a five-position rotary switch when required to attain high temperature, provides very small temperature fluctuations. A warning light to indicate the failure of the fan and a pilot light on the thermostat are fitted on the control panel. The oven is useful for all laboratory purposes and is ideal for the sterilisation of syringes, etc., at 165°C. Its internal dimensions are 17 in. cube.



[Photo: Electricals Ltd.]

Electric oven with close temperature control.

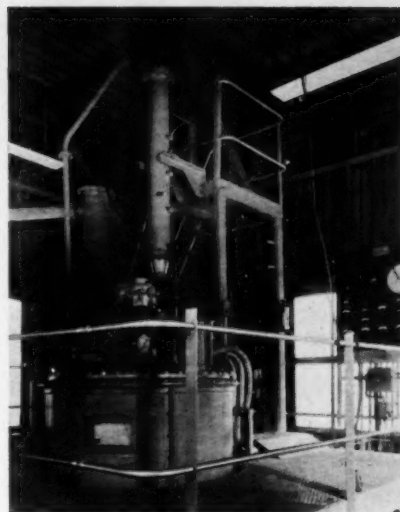
### Isoelectric heating systems

Isoelectric heating plants designed in all sizes from a few gallons up to 2,000 gal. or more are in daily use for the manufacture of such products as alkyd resins, stand oil, varnishes, dehydration of castor oil, oil deodorising, etc. Manufactured by Kestner Evaporator & Engineering Co. Ltd., they are claimed to be economic in use and to have many technical advantages, including no local heating, accurate temperature control, rapid heating, low power requirements, low maintenance and perfect safety.

Each heating element is built in the form of a flat helix and the number of elements depends upon the power supply and shape of the kettle to be heated. The elements are made of nickel chrome alloy of heavy gauge and extremely robust. They are held in the element carrier by heat resisting insulators and the carrier itself is usually made of stainless steel. The electric power supply is fed to the elements by bi-metal conductors. The whole element assembly, including conductors, is suspended from the cover of the kettle so that when the latter is raised the elements are withdrawn with it as a complete assembly.

There is no limitation to the shape of the kettle, but for resin and varnish plants a cylindrical shell with dished bottom and cover is normally employed. The kettles are made suitable for working under vacuum or pressure and are built in stainless steel, aluminium, Monel, copper, mild steel, etc. The kettle is normally provided with a stirrer of the paddle type with one blade below the heating elements and another blade immediately above them forcing the liquid upwards. Stirrers of the vortex, anchor and propeller types can be supplied as an alternative and a foam breaker is a standard fitting on resin kettles.

The kettle is completely enclosed in a heavy gauge steel lagging case, surrounding both the sides and the bottom. The lagging material is of adequate thickness so that external loss of heat is negligible. An air space is provided between the inner wall of the lagging case and the kettle wall. In the bottom of the space, ports are connected to a fan so that cold air can be blown around the jacket and discharged from ducts at the top of the air space when the contents of the vessel are ready for cooling. When more rapid cooling is required the air blasts can be augmented by cold water sprays playing on the wall kettle. As an alternative, water cooling coils are sometimes fitted to the interior of the kettle. The kettle outlet valve is at the bottom and eliminates the formation of



[Photo: Kestner Evaporator & Engineering Co. Ltd.]

A 1-ton isoelectric plant with special condenser system for alkyd type resins.

a stagnant pocket of the charge which is important when the batch is highly viscous as the whole batch is discharged and the bottom of the kettle left quite clean.

The full mains voltage is usually applied direct to the heating elements and a transformer is included for giving a lower voltage to provide reduced input when maintaining temperature. The temperature is thermostatically controlled at the desired value and a safety cut-out is included which acts at a slightly higher temperature.

In some processes it may be required to add to the batch solid or pasty materials, and in such cases a perforated plate is fitted above the heating elements on which the material is retained until dissolved or melted.

### Low-voltage furnace

To overcome the lack of reliability and difficulty of controlling micro-chemical furnaces, a new type has been developed by A. Gallenkamp and Co. Ltd. The furnace has two sections which are constructed entirely without refractories, having a split cylindrical aluminium case with the lower half fixed and the upper part hinged to allow the furnace to be cooled rapidly. The heating elements are heavy nickel-chrome spirals operating at low voltage, which are easily renewable. The furnace cases are polished inside and out to give a high thermal efficiency, and, being almost lagless, heating is rapid and the furnace quickly steadies down to working temperature. Having a lower heat capacity, it can be cooled down rapidly if required by switching off and opening the case. The element surrounds the combustion tube, giving efficient heat transfer. The furnace is complete with a thermostatic heating mortar of improved design and mounted on a control box, which encloses a transformer and ancillary equipment. The whole unit is self-contained.

### New Plant and Equipment

Fill in the Enquiry Coupon on page 394 for further details of the equipment and plant described in

INTERNATIONAL CHEMICAL ENGINEERING.

# World News

## GREAT BRITAIN

### Acetylene anniversary

The British Acetylene Association was founded in 1901, the year in which the first blowpipe to burn a mixture of acetylene and oxygen was being experimented with in France by C. Picard. With the discovery of the means of manufacturing calcium carbide commercially and the application of acetylene, first to illumination, the necessity for framing regulations for safety in storage, transport and the use of carbide, and the apparatus for generating and applying acetylene was appreciated by the founders. The Association was instrumental in founding the first welding schools throughout the country at a time when technical training in engineering processes did not receive full attention, although now the Institute of Welding has accepted responsibility for technical matters such as training and generally as regards the application of welding in industry.

Last month the Association celebrated its fiftieth anniversary with a series of meetings and functions in London, acting as host to a meeting of La Commission Permanente Internationale de l'Acetylene. The event was timed to coincide with the Congress of the International Institute of Welding. Some 40 delegates attended, coming from Austria, Belgium, Denmark, Eire, France, Germany, Holland, Italy, Norway, Spain, Sweden, Switzerland and the U.S.A. Technical meetings were held and the papers presented dealt with the properties and handling of acetylene at high pressures and in the liquid form. The importance of these subjects is emphasised by the increasing demand for calcium carbide and acetylene in the chemical field, where it is used increasingly in the manufacture of acetic acid, synthetic rubber, plastics and fibres, etc.

The President of Honour for the meeting was Mr. E. Stein, President of the Association for 1951.

### Sales of turpentine freed

Retail sales of turpentine have hitherto been restricted to medical, veterinary and pharmaceutical purposes, but as only a very small quantity is involved in retail sales, and as it is from non-dollar sources, the Board of Trade has decided to make it available in future without restriction.

Existing restrictions on supplies of American turpentine and on turpentine for paints and polishes remain unchanged.

### New Shell managing director

Mr. F. J. Stephens has been appointed a managing director of the Anglo-Saxon Petroleum Co. Ltd., and of the Shell Petroleum Co. Ltd., in succession to Sir George Legh-Jones, who retired recently.

### S.C.I. meet in London

Several hundred members and many foreign guests attended the 70th annual meeting of the Society of Chemical Industry held in London from July 9-13. On the scientific side the theme of the meeting was 'Water in Industry,' and this subject was introduced by Mr. H. W. Cremer, president of the Royal Institute of Chemistry. Other papers included 'Availability of Water Supplies,' 'Water and Crop Production,' 'Production of Distilled Water,' 'Corrosion Problems,' 'Water in Brewing,' 'Instrumentation in Water Usage,' and 'The Disposal of Industrial Waste Waters.'

As is usual at S.C.I. annual meetings, there was a big programme of works visits for members. Among the places visited were: the research station of the Anglo-Iranian Oil Co., Sunbury-on-Thames; J. Lyons & Co. Ltd., London; Kodak Ltd., Harrow; Whitbread & Co.'s brewery, London; the Metropolitan Water Board laboratories; the British Drug Houses Ltd., London; Battersea and Kingston power stations; the Chemical Research Laboratory and the National Physical Laboratory, both at Teddington; the Pyrene Co. Ltd., Brentford; I.C.I. Paints Division, Slough; Tate and Lyle's refinery and research laboratory; I.C.I. Plastics Division, Welwyn; the Lafarge Aluminous Cement Co. Ltd., West Thurrock; Bechtel Gas Works; the Fuel Research Station, Greenwich; the Atomic Energy Research Establishment, Harwell; Glaxo Laboratories Ltd., Greenford; and the B.C.U.R.A., Leatherhead.

On the social side there was the annual dinner at the Dorchester Hotel, at which the chief guest was Sir Roderic Hill, Rector of Imperial College, a reception by the University of London, and a reception by Imperial Chemical Industries Ltd.

At the annual dinner honorary membership of the Society was bestowed upon seven distinguished foreign chemists and chemical industrialists, among whom were the Nobel prize-winners, Prof. P. Karrer (Switzerland) and Prof. A. W. K. Tiselius (Sweden), and Mr. W. McN. Rand, a former president of the Monsanto Chemical Co., U.S.A.

At the conclusion of the meeting the new president of the Society, Mr. John Rogers, O.B.E., chairman of Imperial Chemical Industries Ltd., formally assumed his office in succession to Mr. Stanley Robson, who has been president for the past two years.



Mr. John Rogers.

### Sulphur-from-oil plant

A big new plant for sulphur manufacture is to be built at Fawley, near Southampton, by the Esso Petroleum Co. Ltd. Work on the plant is to start soon and when it is completed, early in 1953, it is expected to produce about 12,000 tons of 95.5% chemically pure sulphur p.a. Almost all of this will be available to other industries.

This project forms the third stage of construction at the new Esso Refinery, now being built at Fawley, which will eventually have an annual output of more than 6,000,000 tons of petroleum products. The first two stages comprise the building of the main refinery units for the production of fuels and lubricants.

The new refinery has been designed to extract from crude oil the maximum quantity of sulphur economically available in the refining operation and, at the same time, to use as little sulphur as possible in its various treating processes. In these processes no sulphuric acid whatsoever will be used and for a refinery of such size and complexity, using crude oil from the Middle East, Fawley may well have the lowest sulphur requirements of any in the world.

Between now and Christmas it is expected that the refinery will produce about 2,000,000 tons of petroleum products, including 500,000 tons of motor spirit. When fully completed in 1953 it will employ about 2,500 workers and its output will be about 6,500,000 tons p.a., including 1,750,000 tons of motor spirit.

### Chemical research on view

Open days at the Chemical Research Laboratory of the D.S.I.R. at Teddington have been arranged for 1951 and applications from industrial firms wishing to send representatives should be sent, before August 31, to the Director for the following sessions; September 19 (afternoon), September 20 (morning), September 21 (morning or afternoon). It is not necessary for firms already on the invitation list to re-apply.

### Obituary

We regret to announce the sudden death of Mr. H. C. Mills, managing director of The Morgan Crucible Co. Ltd.

### More chemicals exempt from K.I.D.

The Treasury have made an order exempting the following chemicals from Key Industry Duty between July 4 and August 19: benzyl cellulose, cellulose acetate (where the weight of the acetyl content, calculated as acetic acid, is less than 55½%, but not less than 50%, by weight of the cellulose acetate and not being cellulose acetate which is an ingredient or forms part of a composite article), N: N'-diethyldiphenylurea, glyceryl triacetate, i-inositol, naphthol, sodium 1: 3: 6-naphthalenetrisulphonate, sulphaquinoxaline and 3: 3: 5 trimethylcyclohexyl mandelate.

### New engineering research buildings

The Simon Engineering Group's new research and development buildings were formally opened on July 3 by Sir Henry Tizard, chairman of the Advisory Council on Scientific Policy, at a luncheon to over 300 guests representing the senior levels of the industrial and scientific world and of various Government departments.

Proposing the toast of 'the guests,' Lord Simon of Wythenshawe said in the course of his speech: 'We have spent £250,000 on the new buildings and equipment. In the last five or six years we have achieved about £25,000,000 of exports to something like 40 countries in most parts of the world, including the U.S. and Canada. Our present order book of over £50,000,000 includes a large coke oven battery in America, a £2,000,000 contract for coal preparation plant in Turkey, a £2,000,000 grain elevator contract for the Argentine Government, and power station contracts in Australia and the Middle East for about £6,000,000. We also have a heavy programme at home in building power stations for the British Electricity Authority, coal-cleaning plants for the National Coal Board, coke ovens for new steelworks like Margam and Shotton and many sulphuric acid plants. In connection with the sulphuric acid shortage, Simon-Carves have been appointed main contractors for the United Sulphuric Acid Corporation's £3,500,000 project for plant at Widnes to produce sulphuric acid from anhydrite, and they are acting in the same way for the I.C.I.'s £1,500,000 anhydrite scheme at Billingham.'

Sir Henry Tizard said it was not yet possible to envisage the time when atomic energy would replace coal to a large extent in the manufacturing industries, but it was possible to foresee the time when it would supplement coal in the production of power. Anyone who had seen the modern atomic pile producing heat on a large scale was impressed with the safety and silence and perfect control of the process. The important thing was not the cost of power but the problem of getting enough of it.

### Chemical glassware display

Among exhibits in the chemical glassware section of the British Standards Institution exhibition at the Science Museum, London, was a stand mounted by Quickfit & Quartz Ltd. Features of their exhibit included standard reaction or vacuum distillation vessels, both a plant-scale flask (200 l. capacity) and a laboratory-scale flask (2 l. capacity).

Also shown were standard fractionating or scrubbing column units with Raschig ring packing and a model of a glass plant for production of pure sulphuric acid. The output of this plant is 20 to 25 tons week, and the scale of the model was 1 in. to 1 ft. Also displayed were standard stillheads with provision for temperature reading, and standard heat exchanger units.

### New unit triples oil refinery's capacity

The second distillation unit at the Stanlow (Cheshire) refinery of the Shell Petroleum Co. Ltd. started operation in June, nearly a month ahead of schedule. This trebles the plant's capacity, bringing it to over 3,000,000 tons a year. The refinery, which first came on stream in November 1949, is due for completion in January 1952. In addition to producing a comprehensive range of petroleum products, including petrol, gas oil, fuel oil, synthetic detergents and chemical solvents, it will also make full use of its refinery gases as raw materials for its own large chemicals-from-petroleum industry. Five further units are yet to be completed, including a cat-cracker, on which work is well advanced. These installations will permit a variation in the output of refined products to suit market demands. Some 3,400 men are still employed on the construction of the plant.

Recently, what is believed to be the biggest lifting operation of its kind in the U.K. was successfully carried out at Stanlow. A welded steel absorber column (weight 240 tons, length 170 ft. and diameter approximately 10 ft.) was raised to vertical by huge gantries specially designed for this task. Its size may be gauged from the fact that, on the 225-mile journey from the manufacturer's works (Babcock & Wilcox Ltd., Renfrew, Scotland), it had to be transported in three sections and subsequently assembled and welded on the refinery site.

The new extensions at Stanlow form part of a £30,000,000 post-war refinery expansion programme that Shell has undertaken in the U.K. This includes Stanlow's complementary refinery at Shell Haven, Essex, which is already in full operation, with a capacity of over 2,000,000 tons a year, and a plant at Heysham, Lancashire, with a capacity of over 1,500,000 tons p.a. Another related project is at Pernis, near Rotterdam, Holland, where the refinery's capacity has recently been enlarged, enabling it to process over 4,000,000 tons a year. These four refineries, Pernis, Shell Haven, Stanlow and Heysham, are strategically located to feed the northern European market in the most economical way. Taking into account the refineries in France, Germany and Italy, Shell has a total annual capacity of some 20,000,000 tons in Western Europe.

### Chemical duties cut

Changes in the United Kingdom tariff are to be made on September 1, 1951, as a result of the concessions agreed upon during the recent international conference at Torquay. In most cases the new rate of duty is less than the current rate. As regards chemicals, on and after September 1, the duty on **amidopyrin** will be 10% (compared with 33½% at present), **cadmium sulphide** 20% (33½%), **ethyl p-hydroxy benzoate** 25% (33½%), **α-**

**ethylbutyric acid** 10% (33½%), **methyl p-hydroxy benzoate** 25% (33½%), **di-cyandiamide free** (10%), **propyl p-hydroxy benzoate** 25% (33½%), **precipitated calcium carbonate** 10% (20%), **carbon blacks** (other than from natural gas) except acetylene blacks 10% (20%), **potassium chlorate** 7½% (10%), **titanium dioxide** 15% (20%) and the following natural **essential oils**: **vetiver** (cuscus) and **ylang-ylang free** (10%).

### Heat transfer conference

The Institution of Mechanical Engineers has announced further details of the conference on developments in the mechanism of heat transfer and in the design of the necessary apparatus, which is to be held in London on September 11-13 next. The organisation of the conference is being undertaken jointly by the Institution and the American Society of Mechanical Engineers, with the co-operation of many societies in this country, the British Commonwealth, Europe and the U.S.A. The Institution of Mechanical Engineers and the American Society of Mechanical Engineers are arranging for the presentation of groups of papers, and it is expected that each of them will submit from 40 to 50 separate papers, making a total of from 80 to 100 papers for discussion on the many aspects of the subject of heat transfer. It is proposed to restrict the papers to developments which have taken place during the last ten years, and the following main group headings have been agreed as covering the field of discussion: heat transfer with change of state; heat transfer between fluids and surfaces; conduction in solids and fluids; radiation, instrumentation, measurement techniques and analogies; special problems, such as heat transfer in turbine blade cooling, in liquid metals, gas turbines and piston engines. It is intended that there shall be technical sessions in the morning and afternoon of each day of the conference. They will be held in the hall of the Institution of Mechanical Engineers, Storey's Gate, and the Caxton Hall, Westminster. On Tuesday evening, September 11, there is to be a *conversazione*, and on the following evening it is anticipated that a James Clayton lecture will be delivered.

### International welding congress

About 500 delegates from 21 different countries attended the first congress organised by the International Institute of Welding held last month in Oxford. In addition to discussions of technical papers, the 14 technical commissions reported on their work. Members of the congress also visited the Abingdon laboratories of the British Welding Research Association as well as the Pressed Steel Co. Ltd. and the Cowley works of Morris Motors Ltd. During the second week of the congress visits were made to works and shipyards in all parts of the country.

## Public health engineering

The report on the first year of the new post-graduate course in public health engineering organised jointly by the Imperial College of Science and Technology and the London School of Hygiene and Tropical Medicine indicates that there is a widespread need for the education of engineers on the broad lines which are being followed. At Imperial College, a laboratory has been equipped for the examination of water and sewage and for experiments in treating processes and a public health engineering library is being set up; both facilities were made possible by grants from the Rockefeller Foundation. Also, a number of departments have co-operated in giving courses in a variety of subjects which are normally outside the scope of engineering. At the London School of Tropical Hygiene students have followed a course in occupational health.

## Courses on instrument maintenance

A scheme of courses and examinations in instrument maintenance has been started by the City and Guilds of London Institute to increase the number of mechanics and technicians concerned in the maintenance, repair and installation of the instruments used for process and production control in industrial plants and in the Forces. Particular attention is being given to the requirements of the chemical, iron and steel and petroleum industries, to fuel economy and the needs of the Services. The course of part-time study is of five years' duration, but there is provision for students holding an appropriate Ordinary National Certificate to enter the course at the beginning of the fourth year.

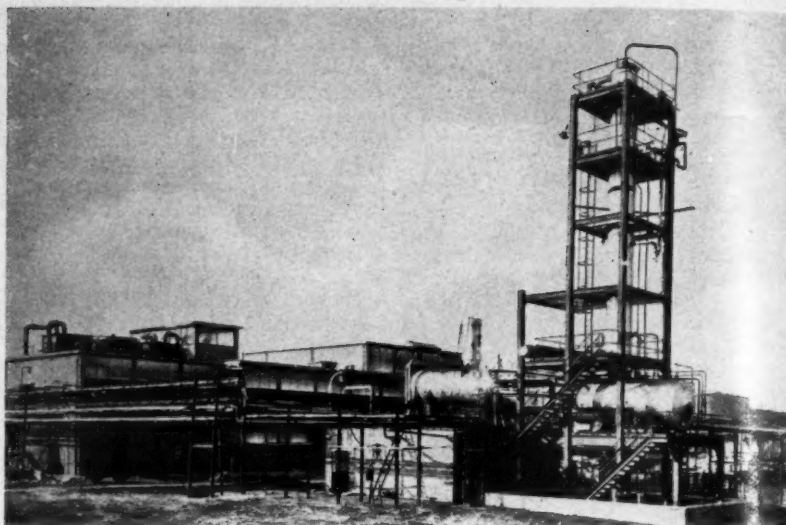
## ITALY

### New zinc manufacturing plant

A new plant for processing zinc ore is being completed at Gorno, Bergamo province. The plant will produce 18,000 tons of zinc ingots a year. By 1952, when the new plant will commence operations, total output of zinc ingots will amount to 70,000 tons p.a. This will leave a large margin for exports, since domestic consumption is not more than 35,000 tons p.a.

### Progress at Condor refinery

Details of the progress on Condor's 1,300,000 tons p.a. refinery at Rho, near Milan, were given at the annual general meeting of Condor S.p.A. per l'Industria Petrolifera e Chimica. The refinery is connected with the central port of Genoa by a 72-mile pipeline with a diameter of 12 in. Purchase of materials for this pipeline and for the associated tank farm at Fegino (Genoa) is almost complete. Construction of some of the refinery plants is in progress and it is expected that the refinery will be in full operation by the end of 1952. Petrocarbon Ltd., of Manchester and London, are acting as consultants to Condor



Preliminary treatment unit of the new Manchester polystyrene plant of Styrene Products Ltd., a company recently formed jointly by Petrochemicals Ltd. and Erinoid Ltd. The plant covers an area of nearly 5 acres, and production capacity is between 5,000 and 6,000 tons of polystyrene p.a. The raw material for polystyrene, monomeric styrene, will eventually be manufactured from materials supplied by the adjacent petroleum chemicals plant of Petrochemicals Ltd. At present imported monomer is being used.

and are responsible for a large part of the design and planning.

The Condor refinery will comprise (1) an atmospheric distillation plant with a working throughput of 30,000 bbls./day of crude oil; (2) a catalytic cracking plant with a throughput of 15,000 bbls./day; (3) recovery, separation and purification plants for the liquid and permanent gases produced by the distillation and cracking plants; and (4) refining plants for liquid gas, gasoline, kerosene and white spirit, together with a blending plant for the addition of tetraethyl lead to the gasoline. A whole range of petroleum products will thus be made available to Italy and Central Europe. Of particular importance are high-octane-number gasolines and liquid gases (C<sub>4</sub>s and C<sub>3</sub>s) for domestic and industrial use. Storage tank capacity amounts to 82,000,000 gal., and exceptional facilities will be provided for the efficient filling of products into road and rail tank cars. A special deep-water dock is being constructed in the central part of Genoa, where the largest ocean-going tankers can berth. Their cargoes of crude oil are pumped to the refinery by pipeline, via the intermediate storage tanks at Fegino, 2½ miles from the dock.

## GERMANY

### New phosphorus plant

The Stickstoffdünger Co. at Knapsack, near Cologne, has received permission from the Allied Military Security Board to install a 10,000-kW electric oven for the production of elementary phosphorus and phosphoric acid. West Germany's annual demand for phosphoric acid is estimated at about 20,000 tons and for elementary

phosphorus between 12,000 and 15,000 tons. The new plant will be able to meet about 50% of domestic requirements for these two products.

Up till now Germany has had to rely on comparatively expensive imports of these materials, particularly from the U.S. and Africa.

### Chemical engineering congress

Over 3,000 persons from Germany and ten other countries attended a congress held in conjunction with the annual meeting of the Chemical Engineering Committee of the Verein Deutscher Ingenieure from May 27-30 at Frankfurt-am-Main. Some 50 technical papers were read dealing primarily with heat exchangers and evaporators, instrumentation, separation of liquids, processing of fine powders, chemical plant construction and corrosion-proof steels. About 180 firms had individual conference stands, facilitating discussions between producers and users of chemical plant.

## FRANCE

### Packaging research association formed

L'Institut Francais de l'Emballage et du Conditionnement has been formed as a non-commercial organisation furthering the interests of producers and processors of all materials used in packaging as well as packaging machine manufacturers and transport organisations. In addition to acting as an information centre for this field, investigations will be made into modern packaging methods and conferences arranged to develop these techniques in France. Present address is 40, Rue du Colisée, Paris.

## NORWAY

### Study of seaweed resources

The Norwegian Institute for Seaweed Research is now mapping the reserves of seaweed along the coast of Norway and also analysing the many varieties of seaweed. Two firms are now using thousands of tons of seaweed a year in Norway for making such products as animal fodder, and various ingredients for puddings, ice cream, etc. Much of the finished products are exported. Many fishermen are collecting seaweed to augment their income and are paid at the rate of about 3d. per kg. for dried seaweed.

## AUSTRIA

### New sulphuric acid plants

The new sulphuric acid plant of the Bleiberg Mining Union at Gailitz, Carinthia, has started production. Capacity is estimated at 15,000 tons p.a. for the time being, but will later be increased to 30,000 tons. The new plant will use Bleiberg zinc blends, Mitterberg copper ores and, if necessary, pyrites.

Orders have already been placed for the special machinery and apparatus for the second sulphuric acid plant in Linz, belonging to the Austrian Nitrogen Works. In the meantime, work has begun on the opening up of the gypsum deposits near the Grundlsee, Salzkammergut, as the new Linz plant will use gypsum as its raw material. However, this plant is not expected to be completed until 1953.

## ECUADOR

### Sulphur mining concession

The sole rights to exploit the Tixan sulphur mines for a period of 20 years have been granted under contract to Chemical Plants Corporation, New York. As part of the agreement the company will construct and operate a sulphuric acid plant in Ecuador to supply the local market. It is also planned to establish carbon disulphide, caustic-soda and rayon plants with United States and Ecuadorian capital, but because the extent of the sulphur deposits has not yet been determined it is not yet known whether all these projects will materialise.

## PAKISTAN

### Paint output increases

Pakistan's annual production of paints and varnishes has increased from 5,000 tons to about 7,000 tons. Two modern factories, opened in Pakistan recently, have brought the total of paint and varnish factories in the country to about one dozen.

Pakistan's requirements of paint and varnish are estimated at about 10,000 tons p.a., and the Government is reported to be considering a plant to encourage the cultivation of linseed with a view to increasing paint production.

## U.S.A.

### International sulphur allocations

The Sulphur Committee of the International Materials Conference in Washington has made the following allocations of crude sulphur for the third quarter of 1951. The Committee intends to recommend by September 10 firm allocations for the fourth quarter.

	In 1,000 Long Tons		
	Allocation	Import quota	Export quota
United Kingdom ..	106.3	105.0	—
Argentina ..	8.2	—	—
Australia ..	31.6	21.6	—
Austria ..	6.5	6.5	—
Belgium and Luxembourg ..	17.8	17.7	—
Brazil ..	13.5	13.5	—
Canada <sup>2</sup> ..	—	—	—
Cuba ..	2.9	2.9	—
Finland ..	4.5	4.5	—
France ..	31.2	27.7	—
French North Africa ..	4.5	4.5	—
Germany ..	15.1	5.1	—
India ..	11.4	11.4	—
Israel ..	1.0	1.0	—
Italy ..	36.0 <sup>1</sup>	—	17.7
Netherlands ..	2.6	0.5	—
New Zealand ..	16.6	16.6	—
Norway ..	6.3	—	18.7
South Africa ..	16.2	16.2	—
Sweden ..	15.1	11.6	—
Switzerland ..	6.8	6.8	—
United States ..	1050.0 <sup>2</sup>	—	250.0
Oil refineries in:			
Bahrain, Lebanon,			
Netherlands Antilles, Trinidad, Indonesia, Persia ..	8.5	8.5	—
Other Countries ..	4.8	4.8	—
Totals ..	1399.9	286.4	286.4

<sup>1</sup> Does not include 10,000 tons of crude sulphur to be exported as refined.

<sup>2</sup> The Canadian allocation is included in the United States figure in column 2.

### Uranium from phosphate

Uranium will be recovered as a by-product in a new \$10,000,000 phosphate plant to be built by International Minerals & Chemical Corporation in Florida. New processes will be used to produce defluorinated phosphate for animal feed and multiple superphosphate for chemical fertilisers. 'Recovery of uranium compounds will be an additional step in these processes,' according to the president of the company.

When the unit goes into operation in about a year, the plant will produce 100,000 tons of defluorinated phosphate p.a.

### Silicone rubber bonded to metals

A method of joining silicone rubber to steel and other surfaces to form a bond stronger than the rubber itself has been developed by the General Electric Co., U.S.A.

A thin, glue-like primer, sprayed or brushed on the metal or glass to be joined to the rubber, is the key to the process. After drying, the surfaces are placed together under light pressure at about 250°F. The primer then forms a strong bond between the rubber and such surfaces as steel, glass, ceramics, aluminium, tin or copper. The bond is claimed to withstand temperatures from -85 to 500°F. In laboratory experiments it has shown a strength of about 700 lb./sq.in. of holding area.

## New catalyst

A new calcium nickel phosphate catalyst which will convert about 92% of butylene into butadiene, compared with about 70% previously achieved, has been developed by the Dow Chemical Co. The extra butadiene produced should substantially aid the output of synthetic rubber, it is thought.

## New rayon fibre

Commercial-scale production is now under way by the du Pont Co. of a new type of viscose rayon fibre, tentatively known as *Fibre E*. Claimed to have unusual crinkling properties, it is said to open up new possibilities of surface and colour contrasts. Production of *Fibre E*, which is obtained from regenerated cellulose, is in progress on a limited scale at Old Hickory, Tennessee. The new type of rayon is stated to curl or crinkle into a fuzzy wool-like fibre upon treatment with a diluted solution of caustic soda. When combined with yarns made from other types of fibres, it gives a carved effect to cut, brushed and loop-pile fabrics, and produces a two-tone colour effect from a single dye bath.

By variations in the filament size of the yarn, in the strength of the caustic soda solution and the density of the fibre used (as well as different pile heights), a wide range of fabrics with various effects can be produced, ranging from soft suedes and velvets and 'nobby' surfaces for upholstery, rugs and imitation fur cloths. The contrast colour effect is achieved by the combination of *Fibre E* with regular viscose process rayon and cotton.

## Cheaper titanium

Horizons Inc., Princeton, New Jersey, and the Ferro Corporation, Cleveland, have formed a new company, Horizons Titanium Corporation, for the purpose of producing titanium metal. They claim to have developed a new continuous process which is cheaper than the present batch method of production.

Neither company will disclose by how much the new process will cut prices. Titanium now costs \$5 per lb. for sponge and \$7 per lb. for ingots. Production of titanium rose from a few laboratory samples in 1946 to 550,000 lb. in 1950.

At the same time as this announcement, the U.S. Navy reported development of a process for producing titanium at \$1 per lb.

## Ethylene oxide by direct oxidation

A new process which is claimed to cut the cost of producing ethylene oxide by 25% has been developed by the Scientific Design Co., U.S.A., and is available for commercial application in industry. This method uses no chlorine at all, as opposed to the chlorohydrin process generally employed which consumes more than 600 tons day of chlorine.

The E.C.A. is contributing \$855,000 towards the installation of one of these

units in France as part of the \$12,000,000 scheme for the expansion of the petroleum chemicals industry at present being undertaken by the Société Naphtachimie (see INTERNATIONAL CHEMICAL ENGINEERING, April 1950, p. 186). The unit will replace a previously planned chlorohydrin unit.

#### New aviation petrol plant

A contract for the design and construction of a sulphuric acid alkylation plant at El Paso, Texas, has been awarded by the Standard Oil Co. to the M. W. Kellogg Co. Cost of the plant will be about \$1,000,000. As part of the current expansion programme at this refinery, the unit will produce high-octane aviation petrol from a mixture of vapours comprising butylene, propylene and iso-butane. Each day it will produce 1,400 barrels of petrol with an octane rating of approximately 100 to 130.

The plant will have the cascade-type reactor and motor-driven agitators for mixing the hydrocarbons with the acid. Construction is scheduled to start in autumn.

#### AUSTRALIA

#### Big new cannery planned

Plans have been announced for a £A5,000,000 food canning, bottling and military ration pack project by the H. J. Heinz Co. Pty. Ltd. Approval has been given by the Victorian State and Commonwealth Governments for an immediate start on the design and erection of manufacturing facilities at Dandenong, 20 miles from Melbourne. The works will employ 800 persons and will be in production in 1953.

A company spokesman said it was estimated that £A2,000,000 would be spent on land, buildings, machinery and equipment, and at least an equivalent sum would be needed for funds to finance inventories, supplies, etc. The company planned to import structural steel from the U.K. or Europe, cement from Scandinavia, machinery from the U.K., Europe, Canada and U.S.A. and steam generating plant from the U.K.

#### NEW ZEALAND

#### New metal products company

A new company to be known as Chromafuse (New Zealand) Ltd. is to be formed shortly in Auckland to exploit a method of giving a number of metals a stainless steel finish. Land has been obtained in Auckland for the proposed factory, plans for which have already been drawn up. The capital of the company will be £75,000.

The process, developed in Britain during the war, consists of fusing a stainless high chromium alloy into the surface of iron and steel products by penetrating the skin of the metal with chromium. The skin then becomes an integral part of the metal and cannot peel off. Metals treated in this way have been shown by official tests to have been made corrosion- and heat-resistant.

#### SOUTHERN RHODESIA

#### New cement works

It is estimated that Southern Rhodesia's demand for cement is growing by 10,000 tons annually and will have reached 50,000 tons by 1955. According to the *Rhodesian Herald*, plans are under consideration for the establishments of two further cement factories.

If drilling of limestone deposits now under way in Shamva proves successful, the Rhodesia Cement Ltd. will recommend the formation of a new public cement company. The sum required for this would be £1,000,000, of which Rhodesia Cement Ltd., would subscribe £175,000.

Another company, Premier Portland Cement Co., is also drilling limestone deposits near Gwelo to investigate their suitability as a source of supply for a projected factory at Salisbury.

#### BRAZIL

#### Penicillin to be produced

Briston Labor Therapeutica, a subsidiary of Briston Laboratories, Syracuse, New York, is shortly to start production of penicillin at a factory in Sao Paulo.

#### CANADA

#### Sulphur controls in force

Canada's Defence Production Department has ordered complete allocation control over sulphur, and no one can buy or sell sulphur without the approval of the Production Department's Chemical Administrator. The announcement indicated that the move was taken to channel supplies first into defence production and then, in order of priority, into essential civilian industry.

#### New chemical plants

The Shell Oil Co. of Canada is to build a \$3,000,000 petroleum chemicals plant adjoining its Montreal East refinery for the manufacture of isopropyl alcohol and acetone. Mr. Ash, president, states that construction will start this summer and that completion is planned for the latter half of next year. The new plant will initially produce some 20,000,000 lb. of the two chemicals annually.

The British American Oil Co. and Shawinigan Chemicals Ltd. recently formed a new company, B.A. Shawinigan Ltd., who are to spend several million dollars on an acetone and phenol plant in Montreal East. Operation of this plant will be connected with the British American Oil Co.'s oil refinery in Montreal East.

#### Largest catalytic cracker

Construction work has started on Canada's largest fluid catalytic cracker by the M. W. Kellogg Co.

The 25,000 barrels/day cracker is part of a modernisation programme being undertaken by Imperial Oil Ltd. at its Sarnia (Ontario) refinery. Kellogg, through its subsidiary, Canadian Kellogg Co. Ltd., is also scheduled to build a 46,500 barrels/day two-stage vacuum and atmospheric distillation unit, as well as a light ends recovery system. When the equipment is on stream, the refinery's capacity will be increased by almost 30% to 71,000 barrels daily.

#### Chemical firm expands

North American Cyanamid Ltd. is to expand facilities at its Niagara Falls and Welland, Ontario, plants to meet increased demands for basic chemicals. A further step in the expansion programme will be the installation at Willow Island, West Virginia, of equipment to increase the company's production capacity of melamine. Current expansion plans involve installation of furnaces, liquid air equipment, ovens and other facilities.

#### International conferences

August 8-15. British Association for the Advancement of Science, annual meeting, Edinburgh, Scotland.

August 22-31. Second conference on Very Low Temperatures, Oxford.

August 29-September 11. Eighth International Congress of Refrigeration, London.

September 3-7. American Chemical Society meeting, New York.

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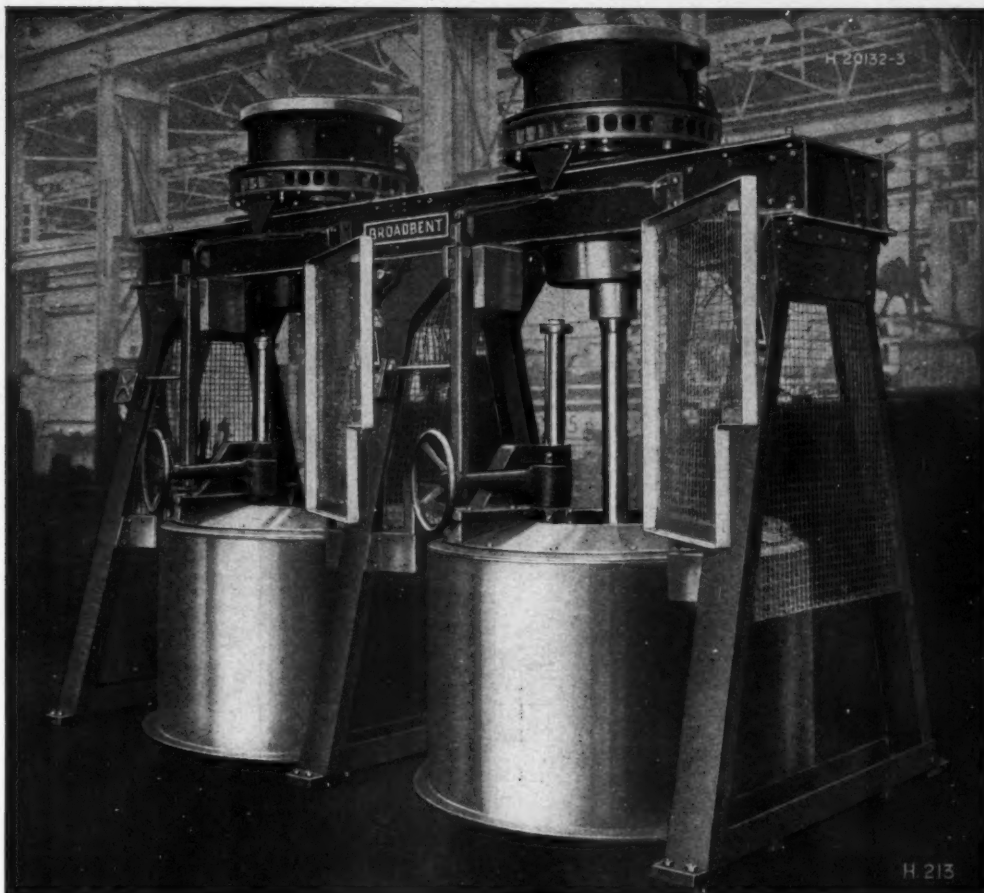
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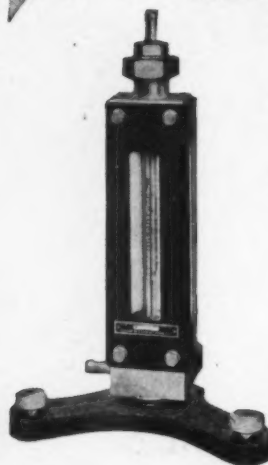
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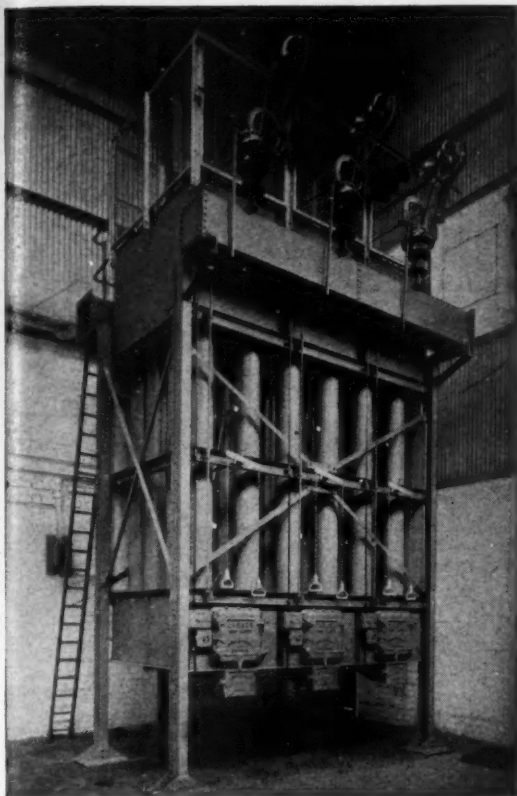
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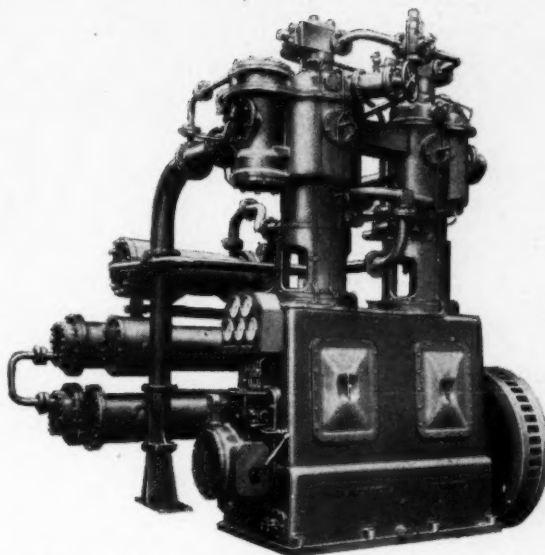
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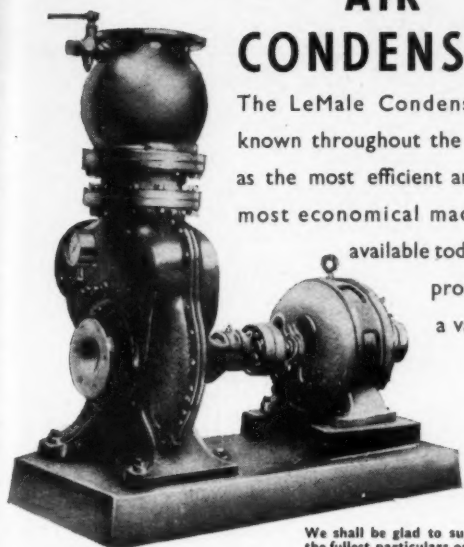
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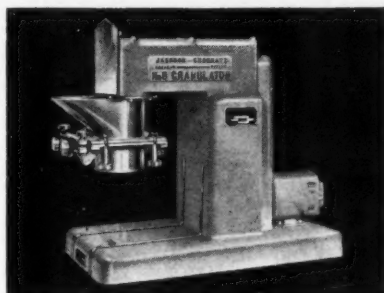
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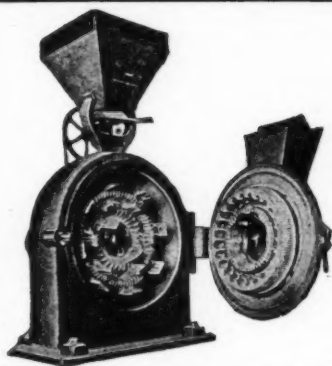
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